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PRACTICAL AND ANALYTICAL CHEMISTRY

AN ELEMENTARY TREATISE
ON
PRACTICAL CHEMISTRY
AND
Qualitative Inorganic Analysis

SPECIALLY ADAPTED FOR USE IN THE LABORATORIES OF
COLLEGES AND SCHOOLS, AND BY BEGINNERS

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PREFACE.

THIS little treatise was commenced to supply a course of Practical Chemistry to my own classes. I was encouraged to proceed with it by finding that a want of a sufficiently elementary and explanatory Laboratory Text-book was very widely felt.

It has been my aim throughout to give all necessary directions so fully and simply as to reduce to a minimum the amount of assistance required from a teacher. The language employed has been rendered simple and intelligible by avoiding the unnecessary use of scientific terms, and by explaining or paraphrasing in ordinary words any such terms when introduced for the first time. The directions how to work, and the descriptions of the preparation and use of apparatus, have been given more fully than is usual, since my own experience, confirmed by that of other teachers, convinces me that one of the most serious hindrances to the utility of many of the smaller Text-books on Practical Chemistry is the too great conciseness of the language employed, which frequently renders it unintelligible to the student unless supplemented by very copious verbal explanation from the teacher.

Whilst making the very desirable amplifications above referred to, the book has been kept within small dimensions, partly by the omission of all such higher instruction as is not required by a student of elementary chemistry, and partly by the insertion of the supplementary or merely explanatory portions in smaller type: the text has been supplemented by appendices containing matter suited to more advanced students. I have also thought it best to avoid entering into any lengthy theoretical explanations. The modern teaching of chemistry is in practice very appropriately divided into two departments—namely, theoretical instruction imparted by lectures or by the study of text-books of theoretical chemistry, and practical instruction imparted in a chemical laboratory by working according to the directions of a practical text-book. Since by this system the student has time and opportunity afforded him for the study of the theoretical and descriptive portions of the science, it is as unnecessary as it is undesirable that his Practical Text-book should tempt him to bestow valuable time in the laboratory upon the study of matters of theoretical—not practical—importance.

The analytical reactions and methods have been carefully worked through from the text by myself and by the members of my classes: the accuracy and intelligibility of their descriptions have thus, I hope, been secured; only those reactions and methods which are commonly employed for analytical purposes have been entered. I have naturally, in selecting analytical methods for an elementary treatise, felt it desirable that those chosen should be as simple and easy of execution as possible; in some cases, however, methods which are most eligible on these grounds have proved on trial to be so inferior in accuracy and delicacy, that they have been abandoned in favour of others which are recommended by their trustworthiness rather than by their simplicity. In such cases, however, I have also described the more simple methods, since they may be employed in analyses in which minute quantities of a substance have not to be tested for. The reactions given in an elementary text-book must necessarily be limited as to number, and I have felt it advisable to introduce such reactions as are useful in general analysis rather than those which claim to be merely interesting and instructive.

The book has been divided into seven sections, the contents of which are fully stated on pages ix.–xv. The first six contain a good practical course for senior students; this may however be modified to suit junior students, or those working with a special object, as is shown in the Introduction.

The seventh section contains full lists of all apparatus, reagents, and chemicals required in working through the different sections; there is also added a list of general apparatus, with a description when necessary of its construction and use. In this section there will also be found full and systematic descriptions of the most simple methods for preparing the different solutions required in analysis, with a statement of the strength most appropriate for each. Experience has proved that these are matters which merit more attention than is usually bestowed upon them. The methods of preparing pure chemicals are omitted, since they may now be readily and cheaply purchased; before using purchased chemicals their purity should, however, always be ascertained by the tests given in this section.

Symbolic notation has been employed, instead of the full chemical names, throughout the sections on analytical chemistry: in its most concise form this chemical shorthand conduces so much to brevity in writing down results that no other plea is required for its use. The simple plan of labelling each bottle in the laboratory with the chemical formula as well as the name of its contents, will prevent any difficulty arising from this general employment of chemical formulæ.

Special features in the book are the arrangement of all Tables *across* instead of *along* the pages; the turning of the book is thus rendered unnecessary—a convenience which will be appreciated by all students of Practical Chemistry.

The "Tables of Differences," which contain for each Analytical Group a summary of the differences of behaviour of its members with reagents, are also special—being an extension of the system employed in Galloway's "Manual of Qualitative Analysis."

It is almost superfluous to mention that free use has been made of the standard works of Fresenius and Rose: much valuable information has been introduced from these sources. I have also frequently adopted the very convenient tabular form of entering analytical methods which is employed in Valentin's "Text-book of Practical Chemistry," and with the author's permission have transcribed, with a few trivial alterations, the excellent Phosphate Table devised by him. My acknowledgments are also due to Dr W. A. Tilden of Clifton College and to the Rev. T. N. Hutchinson of Rugby, and to many other teachers who have suggested valuable improvements.

The book is especially intended to furnish a course of instruction in practical chemistry in the laboratories of our public and other schools. It will thus supply a demand which is rapidly increasing, as the value of a sound elementary instruction in practical science is becoming more widely appreciated, both as a means of mental training and as a preparation for the chemical and medical professions, as well as for many branches of manufacturing industry and enterprise. The fifth section has been inserted for the use of those who are preparing for practical examinations in which proficiency in the analysis of simple salts only is required of the candidate. This is the standard fixed for the Preliminary Scientific (M.B.) Examination of the London University, and for the more elementary examinations conducted by the Oxford and Cambridge Universities, such as those for school certificates.

The sixth section and appendices, however, contain additional details suited for the analytical work of advanced students, and will be found sufficient to qualify a student for the higher University examinations in qualitative analytical chemistry.

The present edition has been subjected to a careful revision, and the analysis of simple salts in Section V. has been recast in a more simple and systematic form.

F. C.

NEWCASTLE-UNDER-LYME, *January* 1880.

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PRACTICAL AND ANALYTICAL CHEMISTRY.

INTRODUCTION.

THE course of practical chemistry which should be pursued by a student depends partly upon his object in studying the science, and partly upon the time and means which he has to devote to the study. The first four sections and the sixth furnish a good general course for a senior student who wishes to obtain a training in practical and analytical chemistry; but for younger students, after completing the first three sections, the most suitable analytical course consists in trying through the reactions in Section IV., and as the reactions for each group are completed, testing several substances containing only one member of the group for the metal present by the table of differences. The analysis of simple substances by Section V. may then be worked through, and, after becoming familar with this, separations of the mixed members in each group are done, leading thus to Section VI. The modification which should be made in the student's course to suit individual cases will, perhaps, be most readily understood by stating the objects of each Section in the book:—

- SECTION I. is adapted to impart experience in chemical manipulation, and to afford practical illustrations of the elementary portions of theoretical or descriptive chemistry; it may be omitted when instruction in analysis only is required.
- SECTION II. is indispensable, and must be carefully perused.

SECTION III. is also indispensable to the student of analytical chemistry, but if the time at his disposal is *very limited*, he may omit the performance of the experiments described in this Section, and merely read through the text.

SECTION IV. must be carefully worked through. A student whose time is limited may, however, simply try the reactions and omit the performance of analyses at the end of each group.

SECTION V. is intended for students whose object is to learn only the analysis of simple salts, such as is required in many modern examinations on practical chemistry.

This section may also be used as an easy beginning in analysis, and as an introduction to more complicated analyses to be made by Section VI., or it may be passed over by the student who is intending to learn general analysis.

SECTION VI. is not required by a student who is learning only the analysis of simple salts, but should be carefully worked through by the student of general analysis, who may, after becoming thoroughly conversant with its contents, pass on to quantitative analysis, or to any special branch of practical chemistry he may require.

SECTION I.

EXPERIMENTS ILLUSTRATING THE PREPARATION AND PROPERTIES OF GASES &c.

Introductory remarks.—In this section full directions are given for the preparation of five gases (viz., oxygen, hydrogen, carbon dioxide, ammonia, and carbon monoxide) and for certain interesting and instructive experiments which may be made with them. The processes of preparation and manipulation required for these gases are more or less typical of those employed for all other gases, and the student will therefore, from the experience obtained by performing the experiments with the above named gases, easily prepare and experiment upon the three other gases (nitric oxide, chlorine, and hydrochloric acid) by following the directions inserted in small print, and any other gas by the account given of it in a treatise on chemistry. Those gases which are in small type may be omitted in the practical course. Two examples of the process of distillation are also appended.

The reference numbers enclosed in brackets refer to the paragraphs which commence with Section II. (p. 27); the numbers will be found in thick type in the text, and at the head of each page the numbers of the paragraphs are also placed in square brackets.

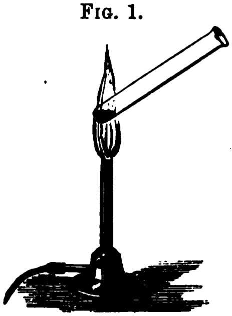
A full list of the apparatus required for this section is given in par. 494, and its use is explained in pars. 1–10 and 14–19, a list of chemicals and certain other requisites will be found in par. 531.

Note.—The student must carefully read through *the whole description* of each experiment before beginning to perform it, and after its successful performance should enter a brief description of it in his note-book.

I. OXYGEN GAS.—When iron is for some time exposed to moist air its surface becomes covered with rust; many other metals undergo a similar change in moist air, but the alteration produced in their appearance is not usually so noticeable as in the case of iron. The liquid metal mercury does not rust as iron does in moist air, but it becomes slowly covered with red mercury-rust when strongly heated for some time in a flask open to the air; this mercury-rust has received the name of mercuric oxide. The fact that metals become heavier by rusting proves that something is added during the process.

EXP. 1.—Place in a clean and perfectly dry test-tube sufficient mercuric oxide to cover the bottom; heat the powder (1, 19) as shown in fig. 1, loosely stopping the end of the tube with the thumb. As soon as small drops of mercury form on the sides of the tube, remove the thumb and quickly place inside the top of the tube the burning end of a slip of wood (*e.g.*, the uncoated end of a wooden lucifer match); the flame will be seen to burn more brightly. If, after again heating the powder for some time in the way just described, the slip be introduced into the mouth of the tube immediately after blowing out the flame and whilst there is a spark at its end, the glowing end will be caused to burst into flame. This behaviour with a burning or glowing slip of wood is one of the most remarkable properties of oxygen gas, and we frequently make use of this property as a "test" for its presence. The chemical change which has occurred is thus represented by an equation:—

FIG. 1.



$$HgO = Hg + O.$$

Since by heat, then, we can separate from mercuric oxide mercury and oxygen, we learn that the process of rusting consists in the metal taking oxygen gas from the air, and the increase of weight above referred to is thus accounted for; if all metal rusts could be decomposed by heat we might obtain oxygen from them just as from mercuric oxide. This method of making oxygen is interesting, since it was the first means known of preparing the gas; it is never used nowadays to prepare large quantities of oxygen, since other substances are known which contain a larger proportion of oxygen, and give it off, when they are heated, more easily than mercuric oxide does—substances which are also preferable on account of their greater cheapness. Potassium chlorate is most frequently employed:—

$$KClO_3 = KCl + O_3.$$

EXP. 2.—Place in a clean dry test-tube a little potassium chlorate, and heat it as in Exp. 1. The white salt, after "decrepitating" or crackling, "fuses" or melts, and when further heated, appears to boil; the small bubbles which are given off consist of oxygen gas, as may readily be proved by holding in the mouth of the test-tube a burning or glowing splinter of wood as described in Exp. 1.

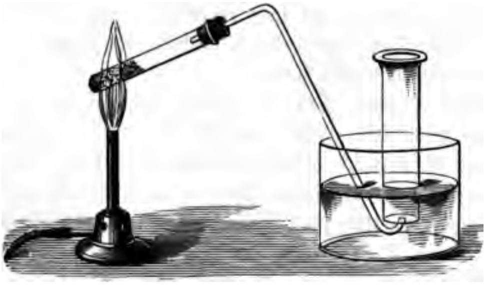
Potassium chlorate gives off oxygen gas much more readily than does mercuric oxide; but if it is mixed with small quantities of certain other substances, which themselves appear to undergo no change, its oxygen is driven off by heat with extreme facility; of these substances, manganic oxide or black oxide of manganese is the one usually chosen.

EXP. 3.—Powder some potassium chlorate (about as much as would fill a watch glass) finely in a mortar, mix with it, by rubbing them together in the mortar, about one-fifth as much powdered manganic oxide, and heat a small quantity of the mixture in a test-tube; the oxygen will begin to come off as soon as the mixture is heated, and a comparatively gentle heat will cause the gas to be rapidly evolved.

In the preceding experiments the oxygen was detected in the tube in which it was prepared, and was allowed to pass away freely into the air. When the gas has to be collected in a vessel unmixed with air, it is made to pass through a bent glass-tube (the delivery tube), which is fitted by means of a cork air-tight into the mouth of the test-tube; the end of this tube dips into some water, and the bubbles of gas are allowed to rise into a vessel full of water and inverted over the end of the delivery tube. This process of "collecting" oxygen is fully described in the following experiment; in the performance of which two students may advantageously work together, one attending to the regulation of heat to the mixture, the other to filling the gas-jars.

Exp. 4.—Select a sound cork, of such a size that, after having been softened by being squeezed or by being rolled with gentle pressure on the floor under the foot, it fits tightly into the mouth of the test-tube to be employed. Then bend (6) a piece of hard glass tubing, about 14 inches in length, into the form shown in the figure; so adapting the bends by trial that when the apparatus is fitted together the bottom of the test-tube may be at a convenient height in the flame, the end of the delivery tube at the same time dipping about a inch under water. Make a hole through the centre of the cork (8), of such a size that the glass tube fits tightly into it. Then test whether the apparatus is air-tight by fitting the glass tube into the cork, and the cork into the test-tube, and blowing down the open end of the delivery tube; no air must be heard to escape, or must be seen to bubble out on moistening the cork; if air does escape, a fresh cork must be taken. Now pour into the perfectly dry test-tube the oxygen mixture (see Exp. 3) off a piece of paper folded into a trough, or scoop up the mixture from the mouth of the test-tube, until the tube is about one-third full, and fit in the cork and delivery tube.

Fig. 2.



Before heating the tube fill the jar in which the oxygen is to be collected with water, close it with a stopper or ground-glass plate or with the hand, invert its mouth in water three or four inches in depth, contained in an earthenware pan or bowl, and carefully remove the stopper or plate. If this operation has been performed with proper precaution the jar will be entirely filled with water, and no air-bubble will remain. Next proceed to heat the upper part of the oxygen mixture, holding the tube in one hand and keeping the lamp slowly moving with the other hand, in order to prevent any part of the glass from being suddenly and strongly heated, which would be liable to crack it. Oxygen gas will soon be evolved, but will not at once appear at the end of the delivery-tube, since it has first to drive out the air which filled the apparatus; as soon as a slip of wood glowing at its end is kindled when held at the mouth of the delivery-tube, the oxygen has driven out the air and is beginning to escape; the end of the delivery-tube is then at once dipped under water beneath the mouth of the jar, and the stream of bubbles rising into it will rapidly displace the water. As soon as the jar is full of gas, close its mouth under water with the stopper or glass plate, and remove it for experiment. The jar may also be removed by slipping under its mouth a small dish or saucer, the water taken out in the saucer then closes the mouth of the jar air-tight.

Precautions.—The water must be removed from the pan, when it rises inconveniently high, by means of a small porcelain dish; if at any time the gas should be given off too rapidly, the flame should be removed until the current slackens; the lower portions of the mixture should be heated only after the upper parts refuse to yield any more gas: when the process is to be stopped, the end of the delivery-tube must be removed from the water before the gas has ceased to bubble out, and the test-tube must not be allowed to touch cold or wet objects, which would cause the hot glass to crack.

Several bottles filled with oxygen will be required for the following experiments, or the same bottle may, if necessary, be refilled with the gas according to the above directions, after the completion of each experiment.

Note.—The use of ground-glass plates, which must close the mouth of the jar perfectly air-tight, is much easier than that of stoppers. An earthenware "bee-hive shelf" which may be used in a common earthen pan, or a "pneumatic trough," is also convenient, since it supports the jar during the process of collection.

Oxygen gas is remarkable for the energy with which it combines with or burns many substances: three examples are given of this property in Exps. 5, 6, and 7.

EXP. 5.—Select a splinter of wood charcoal or a small piece about the size of a nut; the experiment is more brilliant if the surface of the charcoal formed originally part of the bark of the tree. Bind this upon a "deflagrating spoon" with a little fine iron or copper wire; then adjust the wire handle of the spoon in the brass cap, so that when held beside the bottle of oxygen with the cap on a level with the mouth of the jar, the little metal cup is about an inch from the bottom of the bottle. Now heat the charcoal in the Bunsen flame, or better in the blowpipe flame (4), until a part of its surface glows when held in the air, and quickly place it in the bottle of oxygen, with the brass plate covering the mouth of the bottle. (See fig. 7, p. 17.) The charcoal will burn much more brilliantly than in air, throwing off sparks if its surface was "barky":—

$$C + O_2 = CO_2.$$

When it ceases to burn, pour into the bottle a little clear lime-water from a small beaker or test-tube, quickly close the bottle and shake the liquid round inside it; the clear liquid becomes milky, indicating the presence of carbon dioxide gas, as will be hereafter explained.

EXP. 6.—Remove the charcoal from the deflagrating spoon and replace it by a piece of sulphur as large as a pea; heat the spoon in the flame until the sulphur melts and begins to burn with a pale blue almost invisible flame. Then place the spoon in a fresh jar of oxygen, the sulphur will at once burn with a much brighter flame, which emits a beautiful violet light:—

$$S + O_2 = SO_2.$$

Sulphur dioxide or sulphurous anhydride gas remains in the bottle, its presence is proved by its suffocating smell, also by pouring a little water into the bottle and shaking it round. Sulphurous acid is thus formed, and is recognised by dropping into the water a piece of blue litmus-paper, which is immediately reddened, and by pouring in a few drops of red potassium dichromate solution, the colour of which changes to green.

EXP. 7.—Cleanse the deflagrating spoon from any remaining sulphur, and put into it a small piece of phosphorus no larger than a pea. The phosphorus may be cut with a knife; it must be touched only with *wet* fingers, and should be handled as little as possible, since it is liable to catch fire by the heat of the hand; it is always kept under water, being dried only immediately before being used by pressing it between filter-paper or blotting-paper, or with a dry cloth. Set fire to the phosphorus by holding the spoon in the flame, and notice how it burns in the air; then place the spoon in a jar of oxygen—the phosphorus will burn most brilliantly, producing a white substance called phosphorus pentoxide or phosphoric anhydride:—

$$P_2 + O_5 = P_2O_5 .$$

When the phosphorus has ceased to burn, pour in a little water and shake it round in the jar, the white substance dissolves, yielding phosphoric acid:—

$$P_2O_5 + 3H_2O = 2H_3PO_4 ,$$

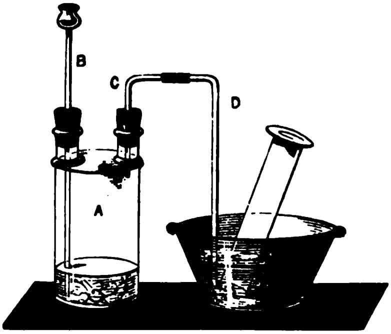
and the water may now be proved to be acid by dropping into it a piece of blue litmus-paper, which will be immediately reddened.

Test for oxygen.—A convenient test for oxygen is to introduce into the gas a slip of wood with a spark at the end, which is caused to burst into flame. Only one other gas possesses this property, and it is readily distinguished from oxygen by other means. This test only detects oxygen when it is in a pretty pure condition.

II. HYDROGEN GAS.—The liquid substance water consists of oxygen gas combined with another gas called hydrogen; several processes are known for preparing hydrogen from water. This gas is, however, most readily obtained from another liquid containing it, called hydrochloric acid, by the action upon it of the metal zinc.

EXP. 8.—Fit a two-necked Woulffe's bottle * (A, fig. 3), with air-tight perforated corks bearing a thistle funnel (B), whose end reaches nearly to the bottom of the bottle, and a tube bent as shown at C and terminating just below the cork: join to this, by means of a short piece of tightly fitting india-rubber tube, a bent delivery-tube D. Pour into the bottle sufficient granulated zinc to cover the bottom, replace the corks, and after ascertaining that the apparatus is air-tight by closing the end of the delivery-tube and blowing down the thistle-funnel, pour in through the funnel sufficient water to cover the zinc and the end of the funnel-tube; then add strong hydrochloric acid gradually until, after mixing the acid and water by shaking the bottle, the hydrogen is seen to rise from the zinc in numerous small bubbles:—

FIG. 3.



$$Zn + 2HCl = 2H + ZnCl_2.$$

Then dip the end of the delivery-tube under water contained in the pan or trough, and allow the gas to bubble out through the water for at least five minutes. This delay is necessary in order to give the hydrogen time to entirely remove the air

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* A wide necked bottle may be used instead, being fitted as shown in fig. 5 (p. 14).

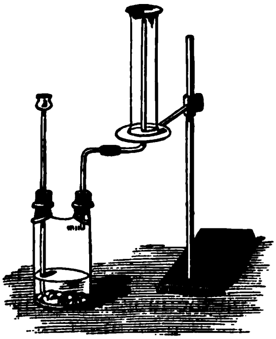
which filled the bottle, and which when mixed with hydrogen produces a dangerously explosive mixture. Before collecting larger quantities of the gas for experiments, ascertain that the hydrogen is no longer mixed with air by inverting a test-tube filled with water over the end of the delivery-tube; as soon as the tube is full of gas close its mouth with the thumb, and hold it to a flame; if the gas burns with a slight explosion, the tube is again similarly filled with the gas and tried in the same way. As soon as the gas burns quietly with a pale flame, a small thick glass cylinder or tube may be filled with it in the same way as was directed for oxygen.

If during the preparation of gas for the following experiments the gas comes off too slowly, it is only necessary to pour in a little more strong acid through the funnel and mix it with the liquid in the bottle by gently shaking the latter.

EXP. 9.—Hold the vessel filled with hydrogen with its mouth open and directed upwards for a short time, the gas will entirely escape; the absence of the gas may be shown by holding a lighted taper in the vessel, when no flame will be seen at the mouth. If the cylinder be refilled with hydrogen and held for a short time mouth downwards, the hydrogen will remain in it, and its presence may be shown by the gas burning with a pale flame when a lighted taper is introduced: these results prove that hydrogen is much lighter than air, since its tendency to rise prevents it from passing out downwards through the open mouth, whilst it readily escapes upwards from the erect cylinder. Since hydrogen is so much lighter than air, it is possible to collect the gas without using water by a process called "displacement." The delivery-tube of the hydrogen apparatus is passed up to the top of the inverted jar containing air, the hydrogen rises to the upper part of the jar and gradually pushes out the heavier air downwards.

EXP. 10.—Fill a jar with hydrogen by "displacement." For this purpose fit upon the india-rubber joint of the hydrogen apparatus a delivery-tube bent as shown in fig. 4, and when a brisk effervescence of gas has been caused by pouring in some strong hydrochloric acid, pass this tube up to the top of an inverted jar, and allow the jar to remain in this position for several minutes; it is best to loosely close the mouth of the jar during this process by letting it rest upon a perforated disk of card-board, or upon the round brass cap of a deflagrating spoon, supported upon a retort-stand ring, or upon an iron tripod-stand, since the entrance of air by "diffusion" is thus almost entirely prevented.

Fig. 4.



Remove this jar, keeping it mouth downwards, and push up inside it a burning wax taper five or six inches in length; the hydrogen will be lighted, and will burn with a pale flame at the mouth of the jar, but the flame of the taper will be seen to be extinguished by the gas: the taper may, however, be rekindled by holding it in the hydrogen flame burning at the mouth of the jar.

Exp. 11.—Cover the bottle and funnel-tube with a cloth, to prevent accident in case of an explosion, and light the hydrogen at the end of the delivery-tube used in the last experiment. Hold over the flame a perfectly clean, dry, and cool tumbler or beaker, the inside will become dimmed with moisture, showing that hydrogen gas burning in the air produces water:—

$$H_2 + O = H_2O.$$

Note.—Since all gases which have been in contact with water contain more or less vapour of water, or are "moist," it is usual to employ for this Exp. a stream of hydrogen gas which has been freed from moisture or "dried." A gas is dried or "desiccated" by passing it through some substance which readily absorbs moisture. Strong sulphuric acid (oil of vitriol), calcium chloride, and quick-lime are the desiccating agents most commonly employed. The gas may be made to bubble through strong sulphuric acid contained in a bottle, B (fig. 10, p. 22), connected, as shown in the figure, with the generating apparatus. It may be also dried by passing it through a tube containing fragments of calcium chloride, or of quick-lime (fig. 10*a*), or pieces of pumice-stone moistened with strong sulphuric acid: the moistened pumice may be contained in a U-tube (fig. 10*b*), or in the bottle B (fig. 10, p. 22).

EXP. 12.—Measure the height of a short thick glass cylinder, and divide it into three equal parts by small pieces of gum-paper stuck upon the outside. Fill the cylinder with water, and after inverting it in water, fill one-third with oxygen (Exp. 4), and the remainder with hydrogen (Exp. 8); let the jar stand with its mouth under water for five or six minutes to allow the gases to mix; then apply a lighted taper to the mouth of the jar directed downwards, taking care not to place the fingers beneath it: the gases combine to form water with a loud explosion.

Test for hydrogen.—Hydrogen gas is recognised by burning with a pale flame in air or oxygen, the flame depositing water on any cold object held above it.

III. CARBON DIOXIDE GAS OR CARBONIC ANHYDRIDE.*—When carbon was burnt in oxygen (Exp. 5) a gas called carbon dioxide remained in the jar: the gas may be prepared in this way, but a much more easy method consists in pouring hydrochloric acid upon some pieces of marble:—

$$CaCO_3 + 2HCl = CO_2 + H_2O + CaCl_2.$$

Chalk or limestone may be substituted for marble, but these do not answer so well.

EXP. 13.—Rinse out the apparatus used for preparing hydrogen, and place in it some small pieces of marble; fit into the india-rubber joint a delivery-tube bent at right angles, as shown in fig. 5; then pour through the funnel-tube sufficient water to cover the marble and the end of the funnel-tube, and then strong hydrochloric acid until gas comes off with brisk effervescence. Place the delivery-tube in a jar, with its end nearly touching the bottom; cover the mouth of the jar with a small disk of cardboard which has had a slit cut in it for the delivery-tube or pass the delivery-tube, through the brass cap of a deflagrating spoon, and allow the apparatus to stand for several minutes.

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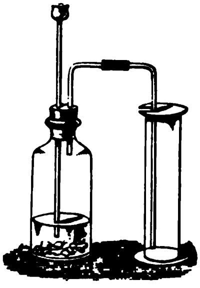
* Formerly called carbonic acid, a name which is incorrect, since all acids contain hydrogen.

Carbon dioxide being much heavier than air, will soon fill the jar by "displacement"; that is to say, it will collect in the lower part of the jar, and, by gradually rising in it, will lift out the air. Since this gas has the property of extinguishing a burning taper, it is easy to ascertain when the jar is full by holding a lighted taper just inside its mouth: if the flame is extinguished, the carbon dioxide has reached the top. Carbon dioxide gas, being very largely dissolved by water, is rarely collected over water, the process of "displacement" being preferable.

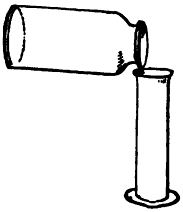
FIG. 5.



EXP. 14.—Allow this jar of carbon dioxide to stand uncovered and with its mouth upwards for a few minutes; then place in the jar a burning taper: the carbon dioxide is shown to be still present in the vessel by the immediate extinction of the flame. Then hold the jar for several minutes with its mouth downwards; on testing with a lighted taper, only air will be found in the vessel. These experiments prove that carbon dioxide is heavier than air, since it remains in a vessel which is open above, and falls out of one which is open below.

EXP. 15.—Since this gas is so much heavier than air, it can be poured from vessel to vessel like water. This may be shown by pouring carbon dioxide from a bottle filled with the gas into a jar full of air, the latter being somewhat the smaller. The bottle is gradually tilted a little beyond the horizontal position, with its mouth over that of the jar. After holding it in this position for a short time it may be proved by a lighted taper that the gas has left the bottle and is present in the jar.

FIG. 6.



EXP. 16.—Pour a little lime-water from a test-tube or small beaker into a jar of carbon dioxide and shake the liquid round in the jar. The lime-water will at once become milky, owing to the lime which is dissolved in the water being converted by the carbon dioxide into chalk (calcium carbonate), which, being an insoluble substance, remains mixed as a white powder or "precipitate" with the water:—

$$CaH_2O_2 + CO_2 = CaCO_3 + H_2O.$$

EXP. 17.—Carbon dioxide gas readily dissolves in cold water, forming a liquid which probably contains carbonic acid:—

$$CO_2 + H_2O = H_2CO_3.$$

The solubility of the gas in water may be proved by displacing the air from a bottle, previously half filled with cold water, by carbon dioxide: then tightly closing the mouth of the bottle with the wetted palm of the hand, and shaking vigorously for a short time: the bottle will adhere to the hand, owing to a partial vacuum being produced by the absorption of the gas by the water. A further proof is afforded by dipping the end of the delivery-tube (fig 5.) to the bottom of a beaker containing water, so as to cause the gas to bubble through the liquid. After the bubbles have passed for several minutes the water may be shown to contain carbonic acid by pouring some of it into a test-tube and adding a little lime-water, which will cause a milkiness*; also, by adding to another part of the carbonic acid solution several drops of blue litmus solution, or dipping into it a piece of blue litmus-paper, which will become red, indicating the presence of an acid: keep this liquid. If this liquid containing carbonic acid be tasted it will be found to possess a taste resembling that of soda-water, and in fact soda-water is merely water which contains a large quantity of carbonic acid, as may be proved by examining it with lime-water and litmus. If some of the water containing carbonic acid be boiled in a test-tube, the carbon dioxide gas is driven off again: the bubbles of gas are seen rising in the water long before the latter boils, and after the liquid has been boiled briskly for several minutes, it may be proved to be free from carbonic acid by giving no milkiness on addition of lime-water, and by not changing the colour of blue litmus-paper or solution. The red liquid prepared by adding blue litmus to the carbonic acid solution will also become blue when it is boiled.

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* Sometimes the milkiness disappears, for reasons explained in Exp. 18 unless much lime-water is added.

EXP. 18.—Dilute some lime-water, contained in a small beaker, with an equal quantity of distilled water, and allow the carbon dioxide gas to bubble through it as in Exp. 17; a milkiness will be produced owing to the formation of chalk (Exp. 16); but if the gas is allowed to bubble for several minutes through the liquid, the milkiness will gradually disappear, since the chalk dissolves entirely in the carbonic acid which is formed by the carbon dioxide dissolving in the water. On boiling some of this clear liquid it again becomes milky, since the carbon dioxide is driven out of the water by heat, and therefore the chalk can no longer remain dissolved. It will be found, on pouring out the water, that part of the chalk remains adhering to the inside of the tube, whence it may be removed by pouring in a few drops of hydrochloric acid. The above experiment explains the origin of the coating or "incrustation" of chalk inside kettles and steam-boilers in which chalk-water is boiled. Such water contains chalk dissolved by carbon dioxide gas present in the water, this gas is driven off when the water is boiled, and a great part of the chalk separates upon the sides of the vessels.

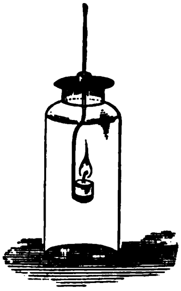
Tests for carbon dioxide.—It is evident that the presence of carbon dioxide gas is shown by its properties of extinguishing a burning taper, and turning lime-water milky: these are the ordinary "tests" for carbon dioxide. We may now proceed to employ them to prove that carbon dioxide is evolved from our lungs during the process of respiration, and also that it is produced by a burning candle.

EXP. 19.—Invert a bottle full of water in a pan of water, and fill it with air from the lungs by blowing the breath out through a glass tube, one end of which is dipped into the water and held beneath the mouth of the bottle. In order to obtain air *from the lungs* a full breath should be drawn, and the nose then closed by pinching it with the finger and thumb: before allowing any breath to pass up into the bottle, the greater portion should be breathed out through the tube so as to replace the air contained in the windpipe, mouth, and tube by air from the lungs, the remainder of the breath is then allowed to bubble up into the bottle. Close the bottle, remove it from the pan, and introduce into it a lighted taper, the flame will be immediately extinguished.

Now blow air *from the lungs*, obtained as just described, through a glass tube into lime-water contained in a small beaker, the lime-water will become milky.

EXP. 20.—Fasten a small piece of candle or wax taper upon the deflagrating spoon, and place it alight in a bottle of air, the mouth of which is closed by the brass plate (fig. 7). After burning for a short time the flame will be extinguished, and if relighted will be again extinguished when placed in the jar. Now pour in some lime-water from a small beaker, and shake it round in the bottle, the liquid will be rendered milky.

FIG. 7.



A similar experiment may be performed, substituting for the candle the flame of coal gas which is burnt from a jet inside the inverted bottle, and closing the mouth of the bottle with a glass plate, as soon as the flame is extinguished. The extinction of the flame, and the milkiness then produced by shaking lime-water in the bottle, will prove the production of carbon dioxide by the combustion.

Hence carbon dioxide gas is constantly being introduced into the air by respiration and combustion, and we should therefore expect to be able to detect its presence in air by the above tests. It is manifestly not present in sufficient quantity to extinguish a burning taper, but the presence of carbon dioxide in air may be shown by lime-water in the following way:—

EXP. 21. Pour some clear lime-water into a watch glass, or better a clock glass, and allow it to stand for a few minutes in the air; a film of chalk will gradually form on the surface, and will be seen as white flakes when the water is stirred.

III. *a. Nitric oxide gas* may be prepared in the apparatus used for making carbon dioxide (fig. 5, p. 14).

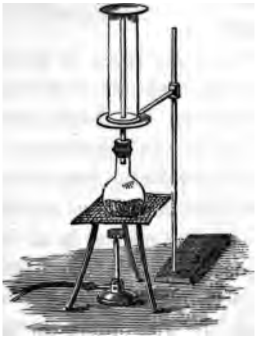
EXP. 22.—The pieces of marble are removed and the apparatus washed out. Some scraps of copper (copper clippings or turnings) are then placed in the flask, and nitric acid diluted with an equal measure of water poured in :—

$$3Cu + 8HNO_3 = 2NO + 3Cu(NO_3)_2 + 4H_2O.$$

A reddish-brown gas soon fills the inside of the vessel, and should be allowed to bubble off for a time through water; it may then be collected over water as was directed for hydrogen gas. Nitric oxide is colourless, but it forms a reddish-brown gas (chiefly nitrogen tetroxide) when mixed with free oxygen. This is shown by filling a jar with the gas over water, and then allowing it to stand with its mouth open in the air, the entrance of the oxygen of the air at once produces red fumes. The appearance of red fumes in the preparation vessel is thus explained, since the vessel is at first filled with air with which the first portions of the gas mingle.

IV. **AMMONIA GAS.**—The familiar smell of common "smelling salts" is due to ammonia gas, which is constantly being given off from the solid "carbonate of ammonia" contained in the bottle. The gas is thus evolved only very slowly, it may be made to come off much more rapidly if the "carbonate of ammonia" is mixed with lime and the mixture is then gently heated : sal ammoniac is usually employed instead of the carbonate of ammonia:—

FIG. 8.



$$2NH_4Cl + CaH_2O_2 = 2NH_3 + CaCl_2 + H_2O.$$

EXP. 23—Powder some ammonium chloride or sal ammoniac in a mortar, and mix with it thoroughly on a sheet of paper about an equal quantity of slaked lime in fine powder. Pour some of this mixture into a small flask (fig. 8) until it is about one-third filled, and close the neck of the flask with a tightly fitting perforated cork, into which is inserted a straight piece of glass tube eight or nine inches long. Heat the mixture gently by placing the flask upon a piece of wire gauze on a tripod-stand and putting underneath it a lighted rose-burner. Ammonia gas will soon be smelt issuing from the end of the tube, and, since it is much lighter than air, may be collected by "displacement," as seen in fig. 8. To ascertain when the vessel is filled with the gas it is only necessary to hold at the mouth of the jar a piece of moistened red litmus or yellow turmeric paper; since ammonia gas changes the colour of the former to blue and of the latter to reddish-brown, it is easy to see whether it has reached the mouth of the jar by observing whether any change is produced in the colour of the paper.

A more easy way to prepare ammonia gas for the following experiments consists in heating a little "Liquor Ammoniæ Fortissima" in the flask (fig. 8) instead of the solid substances.

Ammonia gas does not burn continuously in air at the ordinary temperature, but it burns readily either in strongly heated air or when lighted in oxygen gas. Show this by holding the end of the delivery-tube, from which a stream of the gas is issuing, in the top of a Bunsen-flame; a pale yellowish-green flame of burning ammonia will be seen; and if the end of the tube is dipped into a jar of oxygen, the ammonia may be inflamed as it issues into the oxygen gas.

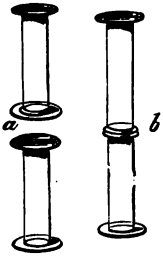
Ammonia should always be collected by displacement, since it is extremely soluble in water, and therefore its collection over that liquid would lead to great waste of the gas. For this reason, also, the vessels in which ammonia is to be collected should always be perfectly dry inside.

EXP. 24.—Place a jar full of ammonia mouth downwards in a vessel of water, and gently shake the jar so as to agitate the water at its mouth; the water rapidly absorbs the gas and rises in the jar to fill the space formerly occupied by the gas.

If a little water be rapidly poured into a jar of ammonia by momentarily partly opening it and at once covering it again with a glass plate or with the hand, and the water be then shaken in the jar, the water, owing to its having absorbed the gas, will, when poured out, be found to have acquired the smell and behaviour with litmus and turmeric papers which characterise the gas. This liquid is in fact weak "Liquor Ammoniæ," a solution prepared in large quantities by letting ammonia gas bubble for some time through cold water. When this solution is boiled the ammonia gas is in great part expelled.

EXP. 25.—Pour into a glass jar a little strong hydrochloric acid, close the jar with a glass plate and shake the acid about inside the jar, hydrochloric acid gas will thus be liberated; the liquid may then be allowed to run out by slipping aside the glass plate for a moment. Place this jar in an inverted position over a jar containing ammonia gas, and covered with a glass plate (fig. 9 *a*), then withdraw the glass plates, so that the mouths of the jars are in contact (fig. 9 *b*), and the hydrochloric acid and ammonia gases can freely intermingle. Dense white fumes of solid ammonium chloride will immediately be formed:—

FIG. 9.



$$NH_3 + HCl = NH_4Cl.$$

This experiment may also be performed by dipping a glass rod into some strong hydrochloric acid, and holding it in ammonia gas as it issues from the delivery tube of the apparatus, or in a jar previously filled with the gas; the same white fumes will at once appear.

Tests for ammonia gas.—Ammonia gas may be readily recognised by its pungent smell; by turning moistened red litmus-paper blue, and moistened turmeric-paper reddish-brown; and also by giving white fumes with a glass rod moistened with *strong* hydrochloric acid.

Gases are frequently made to pass through certain liquids, in order to free them from impurities before they are collected; this is termed "washing" a gas. In some cases gases are purified by causing them to pass over pieces of a solid substance, which absorbs the impurities. The preparation of carbon monoxide gas from oxalic acid will serve to show how these processes are performed.

V. CARBON MONOXIDE.—This gas is produced when carbon dioxide gas is made to pass over red-hot charcoal :—

$$CO_2 + C = 2CO.$$

It is often thus formed in open grates, and is seen burning at the top with its characteristic blue flame. Carbon monoxide is usually prepared by heating solid oxalic acid with strong sulphuric acid, when a mixture of carbon monoxide and carbon dioxide is given off :—

$$H_2C_2O_4.2H_2O + H_2SO_4 = CO + CO_2 + H_2SO_4.3H_2O.$$

EXP. 26.—Place a little solid oxalic acid in a test-tube; pour upon it strong sulphuric acid * sufficient to cover it to a depth of at least half an inch, and heat the mixture. After a short time effervescence will be noticed, owing to gases being evolved; hold in the mouth of the tube a glass rod freshly dipped into lime-water, the drop of lime-water hanging upon its end will become milky, showing that carbon dioxide is one of the gases evolved. Hold a burning taper to the mouth of the test-tube, a blue flame will be seen, caused by the carbon monoxide gas burning in the air.

In order to get rid of the carbon dioxide gas which is mixed with the carbon monoxide, the mixed gases are "washed" with solution of caustic soda, or passed over pieces of solid caustic soda; the soda absorbs the carbon dioxide readily, but allows the carbon monoxide to pass on :—

$$CO + CO_2 + 2NaHO = CO + Na_2CO_3 + H_2O.$$

EXP. 27.—Heat the mixture of oxalic acid and strong sulphuric acid in a flask (A) fitted as shown in fig. 10, and

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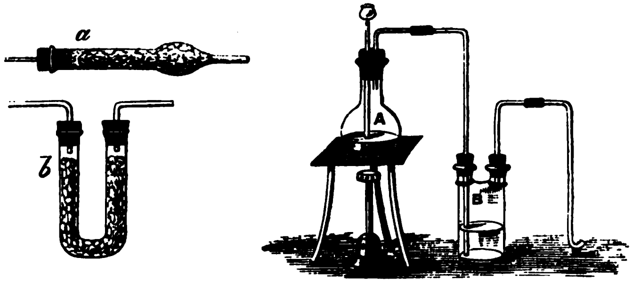
* Strong sulphuric acid is a very corrosive liquid, and great care must be taken not to get any upon the skin or clothes ; should any of this acid or of any other acid get upon the skin it must be at once washed off ; if it should accidentally be spilt upon the clothes, the part must be rubbed with ammonia solution. If the acid has remained for some time on the clothes it will produce a red stain, which will be removed by ammonia solution, unless it has been caused by nitric acid.

pass the gases either into a wash-bottle (a small Woulffe's bottle, or a broad-necked bottle), fitted as shown in B, and containing caustic soda solution; or through a tube (*a*) containing fragments of quick-lime, or a U-tube (*b*) filled with fragments of caustic soda or soda-lime or with fragments of pumice-stone or broken tobacco-pipe stem moistened with strong caustic soda solution. If the carbon dioxide is to be completely removed, the gases must be passed through two or more such tubes or bottles. The carbon monoxide gas thus more or less perfectly freed from carbon dioxide may be collected over water, and will be found to give either no milkiness with lime-water, or a slight milkiness if the stream of gas has been so rapid that the caustic soda has not been able to absorb the carbon dioxide completely. By heating a formate with strong sulphuric acid carbon monoxide alone is given off, and is thus readily obtained free from carbon dioxide.

FIG. 10.



Carbon monoxide resembles hydrogen in being inflammable and in extinguishing a burning taper; it also explodes when mixed with oxygen or air, hence before collecting a cylinder of the gas for experiment, ascertain that the gas coming off from the apparatus is free from air by collecting a small test-tube full and proving that it burns quietly.

EXP. 28.—Push a burning taper up into a cylinder filled with carbon monoxide, the gas will burn with a blue flame at the mouth of the jar but the taper will be extinguished. As soon as the gas has ceased to burn inside the cylinder, pour in a little lime-water and shake it about; the liquid becomes milky, showing that by the combustion of carbon monoxide in the oxygen of the air carbon dioxide gas is produced:—

$$CO + O = CO_2.$$

Tests for carbon monoxide.—Carbon monoxide is recognised by burning with a pale blue flame in the air, producing carbon dioxide which renders lime-water milky.

V. *a. Chlorine gas* may be made in the apparatus employed for the preparation of carbon monoxide: the washing-bottle may either be dispensed with or may be used containing a little water to free the chlorine from hydrochloric acid gas.

EXP. 29.—Place some manganic oxide powdered, or better in small lumps, in the flask A (fig. 10, p. 22), pour upon it some strong hydrochloric acid mixed with about one-third its measure of water, and heat gently in a draught-cupboard or in the open air. A greenish-yellow gas is evolved, which may be collected by displacement like carbon dioxide, since it is much heavier than air:—

$$MnO_2 + 4HCl = Cl_2 + MnCl_2 + 2H_2O.$$

The gas has a very destructive action on the lungs, and must on no account be inhaled; it is usually recognised by its yellowish-green colour, its peculiar smell, and by its property of bleaching moist vegetable colours. This last property is shown by placing in a jar of the gas a piece of moistened litmus-paper or fabric dyed with madder ("Turkey red"): the colours of both will be destroyed. A burning wax-taper plunged into a jar containing chlorine continues to burn with a very smoky flame: oil of turpentine, introduced into the gas by moistening a strip of filter-paper with the warm liquid, catches fire of itself and gives rise to dense smoke. A piece of dutch-foil or copper-leaf also burns when dropped into chlorine.

Tests for chlorine.—Chlorine gas is recognised by its yellow colour, its smell, and its power of bleaching moistened litmus-paper.

V. *b Hydrogen chloride, or hydrochloric acid gas.*

EXP. 30.—Place in the cleansed flask A (fig. 10), which was used for the preparation of carbon monoxide, some lumps of sodium chloride (common salt) obtained by breaking up a mass of melted table salt, or by breaking a piece of rock-salt; pour upon it strong sulphuric acid and heat gently. Hydrochloric acid gas is evolved:—

$$NaCl + H_2SO_4 = HCl + NaHSO_4,$$

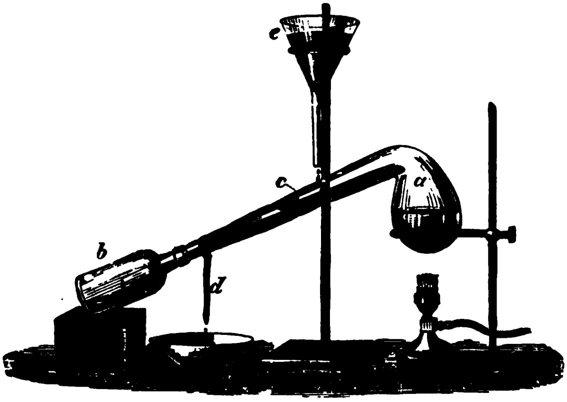
and being heavier than air, may be collected by displacement in the same way as carbon dioxide. The gas fumes strongly in moist air, turns moistened blue litmus-paper red, dissolves easily in water (EXP. 24) giving an "acid" liquid (hydrochloric acid), which, like the gas, turns blue litmus red.

Tests for hydrochloric acid.—This gas is known by fuming in the air, turning moistened blue litmus-paper red, giving white fumes with ammonia gas, and yielding when dissolved in water a milky liquid on addition of silver nitrate solution, which does not become clear on adding nitric acid.

VI. DISTILLATION.—This process is employed to separate liquids which boil at a comparatively low temperature, either from solids, or from other liquids which are not converted into vapour at all or only at much higher temperatures. It consists in boiling the liquid and cooling or "condensing" the vapour, which is thus given off, again into a liquid—the "distillate"; the non-volatile solid or liquid substances present are thus left behind in the vessel in which the liquid is boiled. As examples of this process, the purification of common spring-water from the solid substances dissolved in it, and the preparation of nitric acid, may be performed.

EXP. 31. *Distillation of water.*—Pour into a clean retort *a* (fig. 11) some tap-water through a funnel placed in the mouth, or in the "tubulure" (*i.e.*, the opening for the stopper or cork) if a tubulated retort is employed. Support the retort, whose bulb has been about half filled with water, in a retort-stand or upon a tripod, with its neck sloping downwards and dipping into a small clean flask or bottle *b*, which is partly immersed in cold water contained in an evaporating basin. Cover the bulb of the flask with a broad strip of filter-paper whose ends dip into the water contained in the dish; or, instead of cooling the flask cool the neck of the retort, by wrapping round the lower part of it a piece of filter-paper *c*, and round this a piece of wet string or tow *d*; then arrange a funnel *e* with its mouth partly stopped so as to drop cold water on the upper part of the filter-paper; this water will be drained off by the string *d*, and must not be allowed to run into *b*. On carefully boiling the water in the retort, steam passes into the flask and is there condensed to "distilled water." The first few drops should be thrown away, as they are apt to be impure from rinsing the retort neck and flask. Whilst this process of distillation is going on, add to some tap-water contained in a test-tube several drops of nitric acid and some silver nitrate solution, and notice that the water becomes milky; a separate portion in another test-tube will also be found to become milky on addition of some ammonia and ammonium oxalate solution. These changes are due to the presence in the water of certain solid substances dissolved in it. If the distilled water from the flask be examined in the same way it will remain clear, showing that these substances have been removed by distillation.

FIG. 11.



Tests for distilled water.—Good distilled water should remain quite clear when to separate portions of it are added solutions of ammonium oxalate, silver nitrate, barium chloride, and ammonium sulphide: these tests prove the absence of calcium, chlorides, sulphates, lead, and iron respectively. It should also leave no residue when evaporated.

EXP. 32.—*Nitric acid.*—Clean the retort from Exp. 31 by rinsing out the bulb with a little dilute hydrochloric acid, and then thoroughly with water; let it drain for a few minutes, and place in it some solid potassium nitrate (nitre); then pour upon this, through a funnel placed in the neck or tubulure, a quantity of strong sulphuric acid about equal in weight to the nitre used, and allow the acid to drain off the neck, in case a non-tubulated retort is employed, by supporting it for some time in an upright position. Proceed to distil just as in the preceding experiment; a yellow oily liquid will trickle down the neck of the retort into the flask; this liquid is somewhat impure nitric acid, and will be found to fume strongly in the air:—

$$KNO_3 + H_2SO_4 = HNO_3 + KHSO_4.$$

Test for nitric acid.—Place some small pieces of copper in a test-tube, pour upon them a little of this nitric acid; on warming gently reddish-brown fumes will appear in the tube, and a blue solution will remain (See Exp. 22, p. 18). This property of giving red fumes when treated with copper is often used as a test for nitric acid.

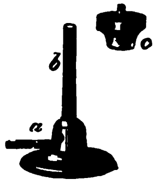
SECTION II.

PREPARATION AND USE OF APPARATUS REQUIRED IN ANALYSIS.

Notes.—Refer to the list in paragraph (494)[*], which shows what apparatus is required for each student, and mark all apparatus as directed in the note at the end of the list.

1. *The Bunsen-burner.*—By far the most useful lamp for general heating purposes is the Bunsen-burner (fig 12). It is a gas lamp so constructed that coal gas entering through the tube *a* is mixed with a proper proportion of air which flows in through the holes at the foot of the burner, and the mixture of gas and air is burnt at the top of tube *b*. The oxygen of the air, which is thus mixed with the gas, burns the carbon in the interior of the flame. Accordingly the luminosity, which is believed to depend upon the existence in the flame of unburnt carbon or carbon-compounds, is destroyed. The Bunsen-flame is therefore useful because it deposits no soot upon a cool object which is being heated in it, and also because it is much hotter, owing to the more complete combustion of the gas, than any of the ordinary flames. Its high temperature, non-luminosity, and colourless appearance also render it very valuable for producing flame colorations, as will be seen hereafter. The lamp should be provided with some means for partly or entirely closing the air-holes when requisite; this is usually

FIG. 12.



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[*] Numbers occurring in the text which are enclosed in brackets refer to the paragraphs commencing in this section. For convenience in reference, paragraph numbers are printed in thick type; and at the head of each page the numbers of the paragraphs it contains will be found enclosed in square brackets.

effected either by a loose perforated ring which is slipped over them, or by making the tube *b* turn round on its long axis. When a small flame is being employed, the air must be partly shut off, else the flame "burns below."

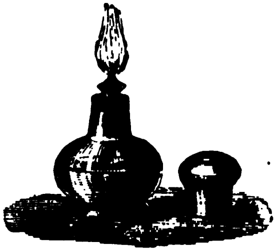
When the lamp is to be used, a piece of tightly-fitting india-rubber tubing 5-16ths of an inch in diameter is pushed over the end of the tube *a*, and the other end of the tubing is slipped over the tube which supplies gas to the working bench, the gas tap is turned on, and as soon as the gas is distinctly smelt issuing from the tube *b*, it is lighted. The flame should be almost perfectly colourless, and give scarcely any light. Occasionally the gas will burn at the bottom instead of the top of the tube *b*; this usually happens when the flame is turned down very low, or the supply of gas is insufficient; also when the quantity of air admitted through the holes is too great, or the burner is lighted too soon after turning on the gas tap. It may be at once detected by looking through the holes in the base of the lamp, when the luminous flame will be seen burning from the small gas jet inside; the flame produced whilst the gas is "burning below" is also characterised by being long and somewhat luminous, and by emitting a very unpleasant smell. In such a case the flame should be at once extinguished by pinching the india-rubber tubing close to the burner, and the gas should be relighted after escaping for a few seconds: the momentary stoppage of the gas-stream by suddenly striking the india-rubber tube upon the bench with the hand will also frequently cause the flame to rise to the top of the burner without extinguishing it. Closure of the air holes serves the same purpose, but this must be done with care, as after the flame has burned below for a short time the metal becomes too hot to be touched. The risk of the gas burning below is prevented by slipping upon the top of the burner a tightly fitting thimble, with a hole in its top somewhat less in diameter than that of the burner.

2. *For diffusing heat over a large surface, the "rose-burner"* is very useful; it is a small perforated metal cap *c* (fig. 12), which, when placed upon the top of the tube *b*,

yields a small circle of flames; the rose-burner is put on and removed by grasping it with crucible tongs; it is of course very hot after being removed, and should never be handled, or placed upon wood, until it is cool.

3. *The flame of a spirit-lamp* is occasionally employed instead of the Bunsen-flame, but for general purposes the spirit-lamp should only replace the Bunsen-burner where coal gas cannot be obtained.

FIG. 13.



The spirit-lamp (fig. 13) consists of a glass vessel containing methylated spirit, into which dips a cotton wick supported by means of a brass, or better, a stoneware wick holder. When not in use the wick should be always covered with the glass cap to prevent evaporation of the spirit. If the spirit is tolerably free from resinous matter its flame will be non-luminous, and will deposit no soot upon a cold object.

4. *The blowpipe* is frequently used to produce a small but very hot flame from the flame of a gas lamp, spirit lamp, or candle, by blowing through it a fine stream of air from the mouth.

FIG. 15.

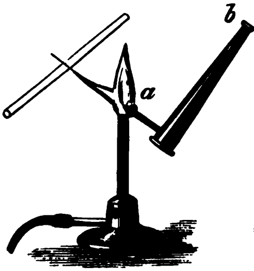
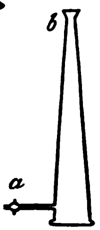


FIG. 14.



In order to obtain the "blowpipe flame" from the flame of a Bunsen-burner, the blowpipe is held by the right hand, with its finely pierced tip *a* (fig. 14), resting on the edge of the burner, and just inside the flame (fig. 15); the mouth-piece *b* is then taken between the lips, and after blowing out the cheeks to their full extent, the air contained in them is forced out through the jet *a*, and causes a small pointed tongue of flame to issue from the side of the gas flame. The chief difficulty in learning to use the blowpipe properly is experienced in acquiring the habit of keeping up the blast of air for some time uninterrupted by the breathing. A little patient trial will, however, soon remove this difficulty, if it is borne in mind that the cheeks must be kept constantly inflated with air, and that the air must be forced through the blowpipe *by the pressure of the cheeks alone and not by the action of the lungs,* breathing being carried on meanwhile through the nose, and the mouth being occasionally replenished with air from the throat just before breathing out the air from the lungs through the nose.

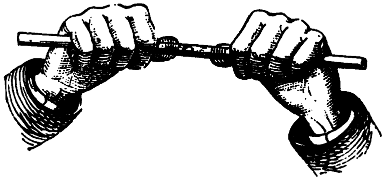
It is frequently necessary to have both hands free whilst using the blowpipe; this may be secured by resting the jet *a* on the top of the burner, and supporting the other end *b*, by the lips alone, as shown in fig. 15; or the blowpipe may be rested on a support of convenient height, such as the ring of a retort-stand properly adjusted.

The bright flame obtained by nearly closing the air-holes of the burner is much better suited for use with the blowpipe than the ordinary non-luminous flame. A burner with an elliptical orifice which gives a flat flame is commonly substituted for the ordinary round flame of the Bunsen-burner; it is easily obtained by slipping a brass tube down the tube of the burner.

A further account of the uses of the blowpipe is give in par. 30.

5. *Glass tube or rod is cut* by laying it upon a flat surface, and making a deep scratch with the edge of a three-cornered file at the point to be cut. The glass is then held with both hands, one on either side of the scratch and close to it, and a gentle pressure is exerted upon the glass as if trying to break it across (fig. 16). If the file mark has been made sufficiently deep, the glass will readily break at the scratch; the

FIG. 16.



sharp edges of a rod or tube should always be at once rounded by holding them in the Bunsen or blowpipe-flame until they are partly melted, or by rubbing them with the face of a file.

6. *Glass tube is bent* by holding the part to be bent in the upper edge of a common fish-tail gas flame, so as to heat at least two inches of the glass (fig. 17). The Bunsen-flame must never be employed for bending glass tubing; it produces a most unsatisfactory bend. The tube is supported by holding it with both hands, one on either side of the flame, and whilst being heated it is constantly turned slowly round on its axis so as to heat all sides equally. As soon as the glass is felt to be soft and pliable, it is taken out of the flame and quickly bent to the required angle. The heated part must not be allowed to touch any thing until it is cold; the soot is then removed from it by a cloth or piece of paper. A bend, if properly made, should be a curve, and should not alter the bore of the tube (fig. 17 *a*); if a sharp angle is made, the bore will be narrowed, and the bend, besides being unsightly, is very liable to break under a small strain (fig. 17*b*.)

FIG. 17.

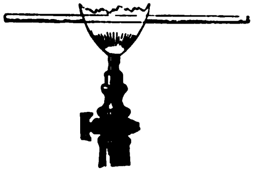


FIG. 17 *a*.

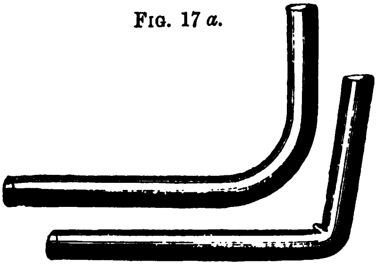


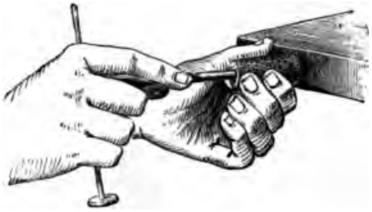
FIG. 17 *b*.

Glass rod may be bent in the Bunsen or blowpipe flame.

7. *Glass tube is drawn out* by holding it with both hands as for bending; and whilst turning it constantly round on its long axis, the part to be drawn out is strongly heated in the Bunsen, or better, in the blowpipe flame (see fig. 15); when the glass is well softened, the two ends are slowly pulled asunder in opposite directions.

8. *Corks are bored* usually by means of brass cork-borers which are used as punches. A borer is selected of the same size as the glass tube which is to be inserted into the cork, rather less than greater; the cork is then pressed against a wooden surface (best against the upright edge of a thick bench or table), and the perforation made in it by gently pushing the borer through whilst constantly turning it upon its axis (fig. 18). Caution and practice will enable the student to make a clean straight hole without damaging the surrounding parts of the cork. In boring a single hole through a cork, the easiest way to make it straight is to bore from the centre of one end halfway towards the other, then reverse the cork and bore a hole to meet this from the centre of the opposite end.

FIG. 18.



A perforation may also be made by pushing a sharply pointed round file carefully through the cork.

The round file is required for smoothing the interior of holes made by the cork-borer, or for slightly enlarging them when they are too small; whilst doing this great care must be taken to leave the hole round in shape, and not to enlarge it so much that the glass tubing when inserted fits loosely.

In perforating india-rubber stoppers, the borer used must be sharp, and must be kept well wetted with water, or better with solution of caustic soda or potash.

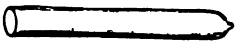
The edge of the cork-borer is sharpened when necessary by rubbing the outer part of the edge obliquely with the face of a fine-toothed three-cornered or flat file.

9. *Sulphuretted-hydrogen tube.*—A tube of the requisite shape will have been used for the experiments in Section I.; it is seen as a bent delivery-tube in fig. 5 (p. 14). If not already made, bend a piece of glass tubing (6) ten or twelve inches in length at right angles into the shape shown in fig. 17 *a*, the shorter limb being about two inches long. This tube is to be kept for passing sulphuretted hydrogen gas through liquids.

10. *Small ignition-tubes.*—A piece of hard glass tubing*, perfectly dry inside and out, and not much less than the eighth of an inch in internal diameter, is drawn out at its middle point by heating it strongly in the blowpipe flame, as shown in fig. 15, p. 29. The narrowed portion of the tube (fig. 19) is then cut across (5) at its middle point, and by heating the conical part *a*, the narrow tube may be drawn off, and a small closed tube is obtained (fig. 19 *a*). If the closed end is strongly heated in the blowpipe flame and blown into whilst hot, it may be expanded into a small bulb, such as that seen on p. 115. Small test-tubes, three inches long by half an inch in diameter, serve well for ignition tubes.

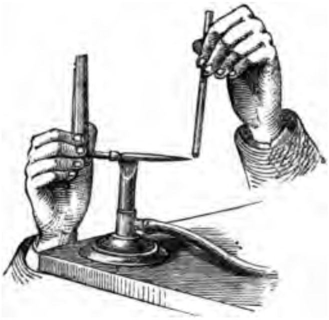
FIG. 19.

FIG. 19 *a*.



11. *Glass stirring-rods.*—Three or four glass rods are made by cutting (5) a length of solid glass rod into pieces, some about seven inches long, others of about half that length. Any small projections are filed or chipped off, and both ends of each rod are then rounded by holding them in the upper part of the Bunsen flame, or better in the tip of the blowpipe flame, and turning the rod constantly round on its long axis until it becomes red-hot at the end (see fig. 20); the sharp edges are thus partially melted. The end of the rod must not be allowed to touch anything until it is cool.

FIG. 20.



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* To distinguish hard glass from soft (lead) glass, hold the tube towards the light with one end near the eye: with hard glass the ring of glass will appear green or colourless, with soft glass black or dark.

11 *a*. Sometimes a very thin glass rod is required; this may be made from a stouter rod by softening it in the blowpipe flame (fig. 15, p. 29), and then drawing it out until the required fineness is produced.

12. *Mounted platinum wires.*—The platinum wire used must not persistently colour the flame green. Draw out a piece of glass tube, six or seven inches in length, at its middle point (fig. 15, p. 29) into the shape shown in fig. 19. Cut it across at the middle of the narrow portion. Each piece of glass thus obtained serves for the handle to a wire. Break off the narrow part of the tube until it extends only about a quarter of an inch from the shoulder (*a*, fig. 19), and into the narrow opening insert the end of a piece of platinum wire about two inches in length and bent round at its other end into a circle about the eighth of an inch across by rolling it round a piece of thick wire; then hold the end of the wire surrounded by glass in the Bunsen or blowpipe flame until the glass melts and thickens around it, fixing it firmly when cold (fig. 21). At least two wires should be mounted in this way. They are then rendered less liable to be lost and much more easy to handle. The wire may also be mounted by softening the end of a short piece of glass-rod in the blowpipe flame, and sticking the wire into it.

FIG. 21.

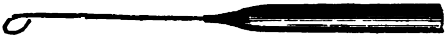
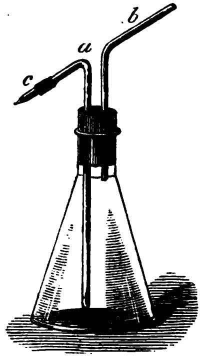


FIG. 22.



13. *Wash-bottle.*—A thin flat-bottomed flask is chosen, best of the triangular shape shown in fig. 22, and of 16 or 18 ounces capacity; the neck must not be less than an inch in diameter. Procure a sound cork which is slightly too large to enter the neck, soften the cork by placing it upon the floor and rolling it backwards and forwards under the foot with gentle pressure; when thus softened the cork must fit tightly into the flask. A vulcanised india-rubber stopper is much more durable for this and most other chemical purposes; it is perforated by a sharp well-wetted cork-borer, or by a wetted round file. Two pieces of glass tubing rather longer than would be required for the tubes *a* and *b* are then bent (6) into the form shown in fig. 22. The ends of the tubes are, if necessary, cut off to the right length, and their sharp edges are rounded by holding them in the Bunsen flame or in the tip of the blowpipe flame, or by rubbing them with the face of the file.

Two parallel holes are then bored in the cork by means of a round file or with a proper sized cork-borer (8); the holes must be somewhat smaller than the glass tubes, and must not run into one another or to the outside of the cork. They are slightly enlarged, if necessary, by the round file. Into these holes the tubes *a* and *b* are then gently pushed with a twisting motion; if the holes have been made of the proper size the tubes must enter somewhat stiffly, but without requiring much pressure. If the holes have been carelessly made too large, the tubes may often be made to fit by slipping upon them little pieces of narrow india-rubber tubing, or by putting the pieces of india-rubber tubing into the holes in the cork before fitting in the glass tubes.

Upon the upper end of *a* is fitted a piece of small india-rubber tubing about an inch in length, into the other end of which is pushed a short jet (*c*), made by drawing out a piece of glass tubing in the flame (fig. 15, p. 29); its opening may be contracted, if necessary, by holding it *perfectly dry* in the flame for a short time. The neck of the flask should then be bound round with twine like the handle of a cricket bat, or tightly covered with a piece of flannel; this prevents the fingers from being burnt when water has been boiled in the flask and its neck has become heated by the steam.

The wash-bottle, when thus fitted up, is filled about two-thirds with distilled water. Tap-water should never be kept in the wash-bottle.

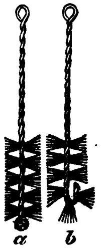
A fine stream of water may then be obtained from the jet (*c*) by blowing down the tube (*b*); this stream serves for washing precipitates and for other purposes. A larger stream is obtained by inverting the bottle, when the water will flow out from the end of the tube (*b*), air entering meanwhile by the tube (*a*).

Hot water is frequently required for washing precipitates; to obtain this the wash-bottle is placed upon a piece of wire gauze supported on a tripod-stand, and is heated by the Bunsen flame, as shown in fig. 10 (p. 22).

14. *Cleaning apparatus.*—It is indispensable, to the success of an analyst that all glass and porcelain apparatus should be scrupulously clean before being used.

Test-tubes, beakers, and porcelain dishes are washed in a stream of tap-water by using the test-tube cleaner (fig. 23 *b*). A little hot hydrochloric acid will remove most stains, but occasionally it is necessary to heat a little strong sulphuric or nitric acid in a vessel in order to cleanse it; hot caustic potash or ammonia solution may also often be used with advantage to remove grease. In fact, in removing a substance from a vessel to which it strongly adheres, the student should always consider what the substance is, and then remove it by a liquid in which it is easily soluble. Each article after having been well washed and rinsed with tap-water, should be placed upside down in the small wicker-basket to drain, or if required for immediate use, it should be rinsed out with a little distilled water. It should be borne in mind that apparatus must be washed as soon as possible after use, as after standing the surface is usually much more difficult to cleanse. The brush must be cautiously moved as it reaches the bottom of a test-tube, since the glass is very thin and the brush is easily pushed through it.

FIG. 23.



Test-tube brush.—It will be found that the piece of sponge at the end of the test-tube cleaner (fig. 23 *a*) is not well adapted to cleanse the bottom of test-tubes and boiling-tubes. A much more efficient end is given to the brush by removing the sponge and bending back the end of the wire stem upon itself at a point just above where the hairs commence (fig. 23 *b*). By slightly curving the part of the stem carrying hairs, the brush will better adapt itself to curved surfaces such as those of porcelain dishes.

Test-tubes whilst in use are placed in the test-tube stand; boiling-tubes may be supported in the wicker-basket, or in a specially made stand. When washed they should be placed to drain mouth downwards in the wicker-basket.

Glass funnels should have their narrow stems cut off to within about half an inch from the shoulder; the sharp outer edge must then be removed by rubbing it with the face of a triangular file. The inside of the narrow neck is best cleaned by washing it with a common tobacco-pipe cleaner, inserted from the shoulder of the funnel so as not to cut the hairs by the edge at the other end of the neck.

15. *Platinum foil and wire are cleansed* by boiling them in hydrochloric acid and rinsing off the acid with water; the wire should then be strongly heated for some time in the blowpipe flame until, on being dipped into pure strong hydrochloric acid, it no longer colours the Bunsen flame. If the tip of the wire cannot be thus cleansed it should be cut off.

Commercial platinum often contains barium, and the wire made from it therefore gives a green colour to the flame; such wire is useless for flame coloration tests and for spectrum analysis.

It is best to keep the foil or wire in a small beaker or dish, containing strong hydrochloric acid diluted with sufficient water to pervent it from fuming; the platinum will thus be ready for use after having been rinsed with water.

16. *Before putting by apparatus* it should be made a rule to wash all glass and porcelain which is not in actual use, and place it in the wicker-basket to drain; the basket is put away with its contents.

All iron apparatus should be carefully dried, and must be kept in a dry place to prevent rusting. *On no account must metal apparatus be kept in the wicker draining basket.*

17. *Heating glass and porcelain.*—A few general precautions should be observed in heating glass and porcelain vessels, to guard against cracking them.

A vessel containing a liquid must never be heated by the flame above the level of the liquid inside.

A dry hot vessel must be allowed to cool before pouring in any liquid, or placing it on a cold surface. See also (18, 19).

18. *Porcelain dishes* are generally used for boiling liquids; they are supported on a tripod or retort stand, and may be safely heated by the naked flame. *Porcelain crucibles* are used for containing solid bodies which are to be strongly heated; they are supported on a pipe-clay triangle placed upon the ring of a retort-stand or upon a tripod-stand: the flame should not be allowed to play steadily at once upon the bottom of the crucible so as to heat it suddenly, but should be constantly shifted by moving the burner, until the porcelain is hot. The crucible should also be allowed to cool slowly on the triangle, as contact with a cold body is very apt to crack it; the crucible and its cover whilst hot are handled by the crucible tongs.

19. *Glass vessels require to be heated* more cautiously than porcelain: a large naked flame must never be allowed to play for any length of time on any one part of the glass surface. In heating a test-tube or boiling-tube, this "local heating" is prevented by holding the tube obliquely with the lower part in the flame (fig. 1) and moving it gently up and down, or by constantly turning it round on its axis. Test-tubes are too narrow for *boiling* liquids in, the liquid being very apt to boil over. *Small* quantities may be boiled and larger quantities heated short of boiling in a test-tube, but the broader "boiling-tubes" are best suited for this purpose. Large glass flasks, such as the wash-bottle, are most safely heated by placing them on a piece of wire gauze on a tripod-stand (fig. 10, p. 22) and heating with a rose-burner; in some laboratories a sand-bath is available, the flask is then heated by being placed on the surface of hot sand. Test-tubes, if not full of liquid, can be held by the neck whilst being heated, if the tube be held obliquely so that the fingers are not over the flame. All risk of burning the fingers is avoided by bending round the neck of the tube a slip of folded paper or of leather, and pinching the ends together close to the tube.

SECTION III.

ANALYTICAL OPERATIONS.

THE student, before commencing the analytical reactions, must become familiar with the operations which are constantly employed in analytical chemistry. The processes will be most readily and perfectly understood by reading the following descriptions, and performing the experiments given in illustration of them.

20. *Solution.*—Many solid substances, if stirred or shaken with water, gradually "dissolve" in that liquid; salt and alum may be mentioned as examples. Other liquids may be employed instead of water, and if they cause the solid substance immersed in them to become partially or entirely liquid and mingle uniformly with the liquid, they are said to "dissolve" the solid, the liquid thus obtained being called a "solution" of the solid, and the liquid which dissolves the solid being termed the "solvent." Thus water, when shaken with sodium chloride (common salt), dissolves it, yielding solution of sodium chloride; and water is therefore called a solvent for sodium chloride. Further, a solid which dissolves in a liquid is said to be "soluble" in that liquid, if it does not dissolve it is said to be "insoluble."

The process of solution is more rapid when the solid substance is employed in the state of powder than when it is in large lumps, since a larger surface of the substance is exposed to the solvent by the powder; it is also much hastened by heating the solvent, which causes a more rapid circulation of the liquid over the solid, and also frequently increases the solubility of the substance.

Two kinds of solution may be distinguished. "Simple solution" occurs when a substance dissolves in a liquid without alteration in composition, the solution possessing therefore the taste, colour, and other general properties of the solid, and yielding the solid substance again when the solvent is removed by "evaporation." Solution of sodium chloride in water is an example of a "simple solution."

"Chemical solution," on the other hand, is always attended by a chemical change in the substance to be dissolved; and since the solution therefore contains a substance differing in composition from the undissolved solid, the properties of the solution are usually not the same of those of the undissolved solid, and on removing the solvent by evaporation the original substance is not obtained.

In dissolving solid substances in liquids, test-tubes, porcelain basins, or small glass flasks are generally employed.

Exp. 33.—Place a piece of potassium nitrate (nitre) in a small clean beaker, fill the beaker one-third with water*, and stir the solid about in the water with a glass rod; the potassium nitrate will *slowly* dissolve in the water; if the liquid be heated the solution will be much more rapidly effected. Powder another piece of potassium nitrate by crushing it and then rubbing it in a mortar with the pestle; place this powder in a test-tube, pour in water, and heat the bottom of the test-tube; the potassium nitrate will dissolve much more rapidly than before, showing that solution is accelerated by powdering the solid and employing heat. Keep these solutions.

Exp. 34.—Powder a little copper sulphate (blue vitriol) in a mortar, place it in a small porcelain dish, half fill the dish with water, and heat it on a tripod or retort-stand. The *blue* copper sulphate dissolves, yielding a *blue* solution. Keep this solution.

These two experiments are both examples of "simple solution." Exp. 33 shows that a colourless solid gives a colourless solution. Exp. 34 proves that a coloured solid gives a coloured solution. This is generally true, and hence we can often infer the presence or absence of a coloured substance in a solution by merely noting the colour of the liquid. More-over, if a drop of the potassium nitrate solution be tasted, it will be found to possess the same taste as the solid; chemists occasionally rely upon taste, as well as upon colour, when examining "simple solutions."

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* Here and in all subsequent cases *water* must be understood to signify *distilled water*.

Exp. 35.—Place in a test-tube a small piece of calcium carbonate (marble or chalk), pour upon it a little water, and heat the tube; the calcium carbonate will be found to be "insoluble" in water. Add to the water some hydrochloric acid: "effervescence," or escape of numerous small bubbles of gas, will occur; the piece of calcium carbonate will meanwhile slowly diminish in size, and will at last entirely disappear in the liquid if sufficient acid is added.

Exp. 36.—Place in a test-tube a small piece of copper; on warming this with a little water it remains undissolved, but on adding to the water some nitric acid and heating, the copper slowly dissolves giving off red fumes, and will be entirely dissolved if sufficient nitric acid is employed.

Experiments 35 and 36 are examples of "chemical solution"; the calcium carbonate is changed by the hydrochloric acid into calcium chloride, and this substance, *not the calcium carbonate*, remains in solution. The copper is changed into copper nitrate, which is then dissolved by the water. It will be noticed that in both these cases a gas is given off; this is a very usual, but not a universal, effect during "chemical solution": the distinguishing fact is that the solid substance which is to be dissolved has undergone a chemical change in the act of passing into solution.

21. *Evaporation.*—When it is wished to obtain a substance, which is dissolved in a liquid, in the solid condition, the liquid is boiled away as vapour, or "evaporated"; the solid substance is then left behind in the vessel.

Exp. 37.—Pour the potassium nitrate solution from Exp. 33 into a porcelain evaporating basin, place the basin on a tripod-stand and boil it over the Bunsen flame until the water has been nearly boiled away, then make the flame smaller and continue heating until the water has disappeared: the solid potassium nitrate is left in the dish.

In evaporating a solution, a small flame or a rose-burner should always be used towards the end of the process, and the flame should be moved about if necessary, else some of the substance will spirt out of the dish.

Often a part only of the liquid is evaporated for the purpose of "concentrating" the solution, *i.e.*, making it stronger by removing a portion of the solvent. A solution, when sufficiently concentrated, will often form "crystals" of the dissolved substance if allowed to stand till cold.

Exp. 37 *a.*—Concentrate the copper sulphate solution made in Exp. 34, and allow it to cool; if sufficient water has been evaporated it will form crystals. Keep the copper sulphate in the dish.

22. *Precipitation.*—Two perfectly clear and transparent solutions, on being mixed together, often become more or less turbid and opaque, owing to a solid insoluble substance being formed in the liquid. A solid substance so formed in a liquid is called a "precipitate."

Exp. 38.—Pour into a test-tube some barium chloride solution, and then some ammonium carbonate solution; a white precipitate is formed owing to the production of insoluble solid barium carbonate. Keep this test-tube with the precipitate.

In the above instance barium chloride and ammonium carbonate separately dissolve easily in water, but if their solutions are mixed they yield by a chemical change two different substances—ammonium chloride and barium carbonate; the former of these dissolves in water, but the latter (like marble) is insoluble, and therefore remains as a fine powder in the liquid.

Substances are very frequently removed or separated in analysis by causing them to form insoluble compounds or precipitates with other substances which are added for the purpose.

In producing a precipitate, care must be taken that the two solutions are well mixed; this mixture may be effected by closing the mouth of the test-tube with the thumb and several times inverting it, or often by simply warming the bottom of the test-tube in the flame; mixture may also be effected by stirring with a glass rod, or by pouring the liquid from one vessel to another: the last three methods are to be used when the liquid present is corrosive, and therefore must not be allowed to touch the skin. A precipitate which does not form readily is often caused to appear more rapidly on thoroughly mixing or agitating the liquid by the above means, or by heating it.

Precipitates produced with different substances differ much in appearance and properties; hence a precipitate is frequently formed to show the presence of a substance or to separate one substance from another. Substances thus added to produce precipitates are called "reagents."

The appearance of a precipitate is usually described by its colour and its condition: it is "flocculent" if it forms in flock-like masses, "crystalline" if in small particles which are seen to be crystals under a lens or microscope, "gelatinous" if jelly-like, &c. A slight precipitate causes only a "turbidity" in a liquid.

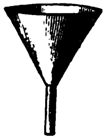
The colour of a precipitate or liquid is often invisible or falsified by gaslight; if the colour is to be seen at night, it should be examined with the light produced by a piece of burning magnesium ribbon.

Occasionally solid substances are used to produce precipitates: thus one metal is not unfrequently precipitated from its solution by immersing in the liquid another metal.

Exp. 39.—Dip a clean penknife blade into some of the copper sulphate solution left from Exp. 37 *a*, to which a few drops of sulphuric acid have been added; after a short time the metal copper is precipitated from its solution and covers the iron as a red film.

23. *Filtration and decantation.*—It is frequently necessary to separate a precipitate from the liquid in which it is suspended. This is effected either by filtration or decantation.

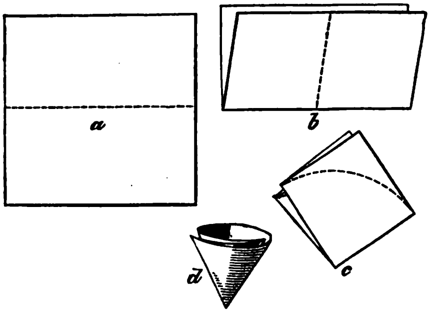
Fig. 24.



23 *a. Filtration* is performed by pouring the liquid containing the precipitate upon some porous paper; the liquid itself runs through the pores of the paper, but the solid particles of the precipitate cannot pass through, and remain on the surface of the paper. The paper employed is called "filtering paper," and the liquid which runs through is termed the "filtrate." A filtrate may frequently be coloured by some substance *dissolved* in it, but it must always be perfectly clear and free from turbidity caused by solid particles *suspended* in it.

Exp. 40.—Measure a glass funnel from its shoulder to its edge along the sloping side (fig. 24), cut a square of filter-paper whose edge is rather less than double this length, fold it across first along the dotted line (fig. 25 *a*), then again as shown by the dotted line in fig. 25 *b*; this gives a square (fig. 26 *c*) which at one angle has four free corners, these are removed by cutting with a pair of scissors along the curved dotted line shown in fig 25 *c*; the "filter" is now made and merely requires to be opened; by separating the edges, formed by cutting off the corners, so that they form a circle, three remaining on one side of the circle and one on the other, a little closed pointed paper bag is formed (fig. 25 *d*); this is gently pressed *with dry fingers* into the *dry* funnel, and the folding altered if necessary till it fits tightly into the glass; it is then moistened all over with a little water from the wash-bottle, and is ready for use.

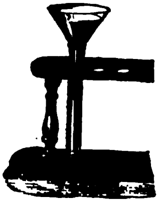
Fig. 25.



The preliminary moistening of the filter-paper must not be neglected, since if the liquid and precipitate are poured upon a dry filter, some particles of the precipitate get between the fibres, which then swell by the wetting, inclosing the solid particles and stopping the pores of the filter.

As an example of the process of filtration, the barium carbonate precipitate obtained in Exp. 38 may be filtered off. Pour the liquid containing the precipitate upon the filter, the funnel being placed with its neck in a test-tube, and the test-tube being supported in a test-tube stand (fig. 26); care must be taken that the inside of the upper part of the test-tube is not wetted, else the space between the neck of the funnel and the inside of the tube is liable to become filled with liquid, and the egress of air being thus prevented the filtration is stopped. The funnel might also be supported upon the ring of a wooden filter-stand, and the filtrate be received in a small beaker instead of in a test-tube. Take great care not to let the level of the liquid in the funnel reach the upper edge of the filter-paper. If the above directions have been carried out, and no hole has been made in the paper whilst preparing the filter, the filtrate will run through perfectly clear leaving the barium carbonate on the filter. Keep the funnel, filter, and precipitate.

Fig. 26.



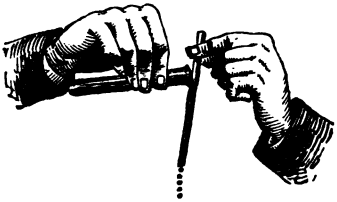
Circular filters can be purchased ready cut, and only requiring to be folded twice to be ready for use. The filter must always be of such size that when placed in the funnel its edge is below the rim. Sometimes the filtrate runs through turbid; it can then usually be made quite clear by passing the whole, or the first portions, of the filtrate once or twice again through the same filter; but with many precipitates a double filter should be used, a double filtration being thus performed in one operation.

It should be borne in mind that hot liquids run through a filter much more quickly than cold; hence, if not inadmissible for other reasons, a solution should always be boiled before being filtered.

Note.—Common filter-paper contains a little calcium carbonate, which is of no importance in ordinary analyses, and is only objectionable when a substance has to be examined for traces of calcium. To remove this impurity in special analyses, the filters are moistened in a funnel with dilute hydrochloric acid, and then well washed with distilled water.

23 *b*. *Decantation.*—Precipitates which settle rapidly and completely in the liquid in which they are formed may frequently be separated from that liquid without the use of a filter. The vessel is allowed to stand at rest until the precipitate has completely settled, and the liquid is then carefully poured off or "decanted" by allowing it to run gently down a wetted glass rod which is pressed against the edge or lip of the vessel (fig. 27), the precipitate being left in the vessel. This process, if carefully performed, effects a fair separation of heavy precipitates. The precipitate obtained by adding sulphuric acid to some boiling solution of barium chloride may be separated by decantation. Keep the precipitate in the test-tube.

Fig. 27.



24. *Washing precipitates.*—A precipitate which has been filtered from the liquid in which it was suspended has often to be washed until it is perfectly free from the adhering liquid: this "washing" is performed as described under 24 *a* if the precipitate has been separated by filtration, and by 24 *b* if by decantation.

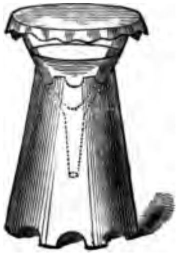
24 *a*. Exp. 41.—Support the funnel, which contains the filter and barium carbonate precipitate from experiment 40, with its neck in a beaker or flask; and blow in a fine stream of hot distilled water from the wash-bottle, so directed by moving the jet with the fingers as to stir up the precipitate well: in this way fill the filter to within a short distance from its edge; let this water run through perfectly, then nearly fill the filter again in the manner just described; repeat this process two or three times, letting the liquid run through perfectly each time before putting in a fresh quantity; the water running through from the third or fourth washing will usually be quite tasteless, and the precipitate and filter will be freed from everything soluble in water. Boiling, or at least hot, water should always be used for washing purposes, as the process is thus rendered much more rapid.

24 *b*. *Washing by decantation.*—Heavy precipitates, after having been separated from liquids by decantation, are washed also by the same process. The precipitate is shaken or stirred with distilled water, and allowed to settle; when the water has become quite clear it is poured off (fig. 27). By several times repeating this washing process with addition of fresh portions of distilled water, the precipitate may be entirely freed from adhering solutions.

The precipitate of barium sulphate, remaining in the test-tube from the experiment at the end of 23 *b*, may be thus washed by decantation.

25. *Drying precipitates.*—A precipitate is often required in a dry condition after it has been filtered off and washed. It is dried by placing the funnel in a hollow tin cone or cylinder, the filter-dryer (fig. 28), and supporting this on a piece of wire gauze upon a tripod-stand over the flame of a rose-burner turned very low; or the filter-dryer may be placed on a gently heated sand-bath. The funnel is thus heated by the current of hot air, and rapidly dries the filter and precipitate. Great care must be taken to regulate the heat and watch the process, so as not to char the filter.

FIG. 28.



A more rapid method of drying a precipitate, after it has drained for a time, consists in spreading the filter upon a piece of wire gauze supported on a tripod-stand; a small flame from a rose-burner is then placed beneath the gauze, and the filter carefully watched to avoid charring it.

A precipitate is partially dried by opening out the filter upon several dry filter-papers: this process may precede those already mentioned.

26. *Removing precipitates from the filter.*—When a small quantity of a moist precipitate has to be taken from a filter to test its behaviour or closely examine its appearance, it is most readily removed by dipping the end of a glass rod into the precipitate; by touching a watch glass or the interior of a test-tube with the end of the rod, a small quantity of the precipitate is deposited for examination.

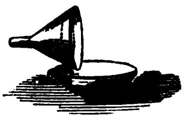
If the precipitate is to be removed from the filter as completely as possible, several methods are available; one or other must be chosen according as circumstances render it suitable.

a. The bottom of the filter may be pushed out through the neck of the funnel with a glass rod, which is small enough to pass easily through the neck (11 *a*), and the precipitate may then be washed down into a vessel beneath with a fine stream of water or other liquid from the wash-bottle.

b. Without breaking the filter the funnel may be held with its neck horizontal, and the rim just inside the edge of a porcelain dish (fig. 29), the precipitate is then washed out by directing a fine stream of water from a wash-bottle against the sides of the filter.

c. The filter and precipitate are allowed to stand for some time, so as to drain off as much water as possible; the filter is then carefully taken out of the funnel, partially dried if necessary by laying it upon several folds of filter-paper, and after removing the portions of the paper which contain no precipitate, together with the empty fold*, it is spread out inside a porcelain dish; the liquid with which it is to be treated is poured upon it, and by shaking the dish so as to cause the liquid to move round and round, and occasionally carefully stirring the precipitate with a glass rod, the precipitate is washed off the paper without tearing the latter to pieces. The paper is then carefully removed by a glass rod.

FIG. 29.



d. If it is undesireable to add a liquid to the precipitate upon the filter, the filter and precipitate, after draining for a short time, are removed from the funnel; the filter is spread out upon a flat piece of glass and the precipitate carefully scraped off with a glass rod or a small spatula. If the precipitate is required dry, the filter, after removal from the funnel, may be carefully opened and spread upon several thicknesses of filter-paper to drain. When there is a large quantity of the precipitate a sufficient quantity may be removed on the end of a glass rod or spatula, without taking the filter out of the funnel. This method is usually the most imperfect, but is frequently the best for other reasons.

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* Filters may be made without this fold, by cutting one of the round papers (fig. 25 *d*) through across one of the folded diameters, bringing the cut edges together, and sharply folding them over several times, pressing them with the nail; if properly made, such a filter is perfectly impervious to the precipitate.

e. A precipitate has sometimes to be dissolved off the filter; the hot liquid used as a solvent may then be poured upon the precipitate, it will run through the filter into a vessel below, taking with it the precipitate in solution: the liquid, after it has run through, should be heated again, and once more poured upon the precipitate, if the latter is not entirely dissolved; this re-heating and returning of the liquid to the filter should be continued as long as anything is dissolved; any remaining portion of the precipitate must then be removed by a little fresh solvent.

f. A precipitate, if small in quantity, may also be rinsed off the filter with the liquid with which it is to be treated or dissolved. The funnel is placed with its neck in a test-tube, the liquid is poured into the filter from another test-tube, and the precipitate is quickly stirred up with the liquid with a glass rod thin enough to pass down through the neck of the funnel; the bottom of the filter is then pushed out through the neck by the glass rod, and the liquid carrying most of the precipitate will run through; if some of the precipitate remains on the filter, the same liquid is poured through the filter again into the other tube, and by thus pouring backwards from one tube to another all the precipitate may be removed.

Note.—The greater part of a precipitate may often be removed by a clean knife or spatula, and the remainder by some one of the foregoing methods.

Exp. 42.—Remove the precipitate of barium carbonate remaining from Exp. 41 from the filter by one of the above processes, using hydrochloric acid as a solvent for method e.

27. *Ignition.*—Solid substances occasionally require to be heated strongly or "ignited." This process of ignition is employed with various objects.

1*st.* In order to see what effect strong heat has upon a substance, since by this means we can frequently detect its composition. For this purpose we use small tubes made from hard glass tubing (10), or small test-tubes.

Exp. 43.—Heat a little mercuric oxide in a small tube of hard glass, after a short time minute shining drops of the metal mercury will be seen on the upper part of the tube; if then a small splinter of wood with a spark at its end be held in the tube, it will burst into flame. The behaviour of this red powder on being "ignited," therefore, shows it to contain the elements mercury and oxygen.

Substances are often heated on platinum foil with the same object. Thus a little charcoal powder when heated on platinum foil burns away.

2*d.*—Ignition is also used as a means of separating from a solid mixture substances which are "volatile," or which change entirely into vapour when heated; these are driven off by heat from other substances which are "non-volatile," or can be strongly heated without changing into vapour, and which therefore remain behind.

Exp. 44.—Heat a small piece of ammonium chloride on a strip of platinum foil held in the flame by crucible tongs; the ammonium chloride being volatile will give off white fumes, which are best seen by removing it from the flame for an instant, and the solid substance will slowly disappear or "volatilise."

Heat in the same way a small piece of potassium chloride; this will melt, but will not disappear, since it is only slightly volatile. It will become solid again on cooling.

Hence, if from a mixture of ammonium chloride and potassium chloride, the potassium chloride is to be obtained alone, the mixture would be strongly heated for some time, when the ammonium chloride would be removed as vapour leaving the potassium chloride behind.

28. *Sublimation* is the process of heating solid substances and converting them into vapour, then cooling this vapour so as to "condense" it or render it solid again. A volatile substance may thus be obtained free from other substances which are not volatile or only slightly so.

Exp. 45.—Heat a small piece of ammonium chloride in a tube of hard glass closed at one end and two or three inches in length; it changes into vapour, which again cools into solid ammonium chloride on the upper part of the tube. This is an example of the process called "sublimation," and the solid substance at the upper part of the tube is a "sublimate."

29. *Fusion.*—Substances are often melted or "fused" together to cause certain chemical changes to occur between them. Melting or "fusion" is performed sometimes on platinum foil, but often in porcelain or platinum crucibles. For fusion in a crucible an intense heat is often requisite; this may be obtained by using the blowpipe flame, but the Bunsen flame will usually suffice if the crucible be surrounded by the conical filter-dryer (fig. 28, p. 47) which steadies the flame and increases the draught.

Exp. 46.—Place upon a slip of platinum foil a mixture of powdered sodium carbonate and potassium nitrate, then a very small quantity of manganese oxide; hold the foil with crucible tongs in the top of the Bunsen flame, or heat the under surface of the foil with the blowpipe flame. The mass will melt or fuse; it is kept in a melted state for a short time and then allowed to cool, when a bluish-green mass is obtained. The production of this colour is due to the presence of sodium manganate formed by the fusion; this colour on fusion with sodium carbonate and potassium nitrate is produced only when manganese is present, and is therefore a test for that element.

30. *Use of the blowpipe.*—The blowpipe is used, as has been already explained (4), to obtain an intensely hot flame which may be made to play in a convenient direction upon any substance to be heated; it is very useful for analytical purposes, since by its skilful use the composition of an unknown substance, when ascertained by other means, can usually be confirmed; and with many substances (especially minerals) a complete, or at any rate sufficient, analysis can be made by the blowpipe alone. The way in which the flame is obtained has been already explained (4).

In a well-formed blowpipe flame two parts may be distinguished: the "inner flame," which is blue and pointed, and around and beyond this an almost colourless flame, "the outer flame." The inner flame is often called the "reducing" or "deoxidising flame," because it separates or "reduces" metals from their oxides and compounds, and in general "deoxidises" substances; the outer flame is also known as the "oxidising flame," because it changes metals into their oxides, and "oxidises" many other substances. These two flames also produce other distinctive effects.

The "inner flame" is best obtained of large size by placing the tip of the blowpipe nozzle only in the very edge of the flame, or even just outside it: the "outer flame" is best obtained by putting the nozzle much further into the gas flame. It may also be noted that the "reducing flame" is furnished most readily by the luminous gas flame, the "oxidising flame" is most easily and perfectly formed from a Bunsen flame which contains some air, and which is obtained by either leaving the air-holes at the foot of the burner entirely open or by only partially closing them.

There are several purposes for which the blowpipe is employed in analytical chemistry with which the student should become familiar; the following experiments will exemplify the most important of them.

31. Exp. 47.—*Borax bead.*—Place a little borax, which has been finely powdered in a mortar, upon a watch glass; moisten the loop of a piece of mounted platinum wire (12) with water or in the mouth, and dip it into the powdered substance, then hold the loop with the adhering powder in the blowpipe flame until the borax melts; if this is insufficient when melted to form a bead which fills the loop, dip the loop with the melted substance upon it again into the powder, more powder will adhere and may be melted in the flame; in this way powder is to be constantly added until when held in the flame a liquid drop or bead is produced which completely fills the loop. This bead must be perfectly colourless and transparent on cooling. Keep the bead on the wire.

If the melted bead easily drops from the wire, either the loop or the bead is too large and must be lessened in size. The most convenient sized loop is one which is rather less than one-eighth of an inch across; it is best made by rolling the tip of the wire round a piece of stout wire. If the bead is too large, remove a portion whilst it is in a fused condition by giving the wire a sudden jerk.

Certain substances when melted in this bead colour it, and as a rule the colour produced by each of these substances has some characteristic tint or peculiarity. The colour of the bead should be examined after it has been heated, first in the outer and then in the inner flame, and must be noted both hot and cold. If the bead appears opaque, on account of too much of the colouring substance having been introduced, it can generally be made transparent by shaking off some of the fused borax, or by flattening the fused bead by gently squeezing it between a small pair of pincers.

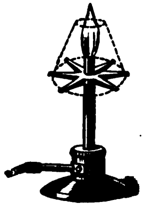
Exp. 48.—Moisten the clear cold borax bead and dip it into some powdered ferrous sulphate, thus taking up *very little* of the powder upon it; then heat the bead and substance for a short time in the tip of the outer blowpipe flame, and examine the colour of the bead by looking through it at a piece of white paper or at a window light; it will be reddish-brown whilst hot, but the colour will become lighter on cooling; now heat the bead again for some time in the tip of the inner blowpipe flame, its colour will be changed to green. These two colours, shown by the borax bead in the outer and inner blowpipe flames respectively, characterise the metal iron.

The fused borax bead is detached by giving the wire a sudden jerk; it may also be removed after cooling by crushing it on a hard flat surface by a sharp blow with the pestle. The wire when not in use should be kept in dilute hydrochloric acid. Should it not be perfectly clean after washing off the acid, the simplest method of cleansing it is to make a fresh bead upon the loop and jerk it off whilst melted, this will usually remove all impurity. A borax bead must never be used for these purposes until it has been found to be perfectly colourless, both hot and cold, when heated in the outer blowpipe flame. If the first bead which is made in the loop is tinted with any colour, it must be jerked off whilst fused and another bead formed with fresh borax, this being also thrown off and replaced if not quite free from colour; this process is to be repeated until the bead is obtained perfectly clear and colourless.

32. *Flame colorations**.—Many substances give a characteristic colour to the flame. The blowpipe flame is often employed to obtain these colours; an ordinary Bunsen flame, however, although it does not give the colours quite so promptly and intensely, answers the purpose well.

The flame best suited for this purpose is obtained by surrounding the Bunsen flame with a conical chimney, reaching about two-thirds of the height of the flame (fig. 30), and then so regulating the supply of air entering into the air-holes that a small luminous point appears near the top of the flame; the platinum wire is held either in this luminous tip or just above it. A filter-dryer also forms a very good chimney, being supported just above the foot of the burner on a wire tripod, which may be made by bending down the ends of a pipe-clay triangle.

FIG. 30.



EXP. 49.—Hold a loop of platinum wire in the inner blowpipe flame, if the wire is clean it will become red-hot without colouring the flame. If it colours the flame, clean the wire by boiling it with hydrochloric acid in a test-tube and then rinsing well with water; or by moistening with strong hydrochloric acid, and heating strongly in the blowpipe flame until it no longer colours the flame. If it cannot be thus cleansed, cut off the end. Now moisten the wire and dip it into a little powdered sodium chloride, or dip the loop into

some sodium chloride dissolved in water so that when removed a drop of the solution remains in the loop; then hold the loop at the tip of the inner blowpipe flame, the outer flame will be coloured bright yellow. Repeat the experiment, placing the moistened loop in the upper and outer part of the Bunsen flame, the same yellow tint will be obtained. This yellow colour is given only by sodium and its compounds.

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* For a more detailed and exact description of flame-tests, see Bunsen's paper in the "Philosophical Magazine" for 1867.

Remove all sodium chloride from the platinum wire loop by heating it strongly in the tip of the blowpipe flame until it ceases to impart any colour to the flame. Then dip the wire into some potassium nitrate solution: this will give to the flame a pale violet colour, which is characteristic of potassium and its compounds.

As will be seen subsequently, the examination of flame colorations, aided by the use of coloured glasses and of a hollow glass prism filled with indigo solution, is of great use it ordinary analysis.

Exp. 49*a*.—Look at the yellow flame coloration produced by sodium chloride through a piece of cobalt glass or through the indigo-prism, no yellow colour will be visible. Now dip the wire-loop into a mixture of a few drops of sodium chloride and potassium chloride solutions, when held in the flame only the sodium coloration is seen, but through the blue glass or prism the potassium is seen to produce a red flame coloration. By means of the blue glass or prism then potassium is discoverable in the presence of sodium, by which its presence is otherwise masked.

By far the most perfect method of examining flame colorations, more especially for the rarer elements or for mere traces of the more common ones, is by the use of the spectroscope; every advanced student should make himself familiar with the use of this instrument, at least in its simplest and smallest form. A paper on the use of the spectroscope for qualitative analysis will be found in the appendix; this is well worthy of the attention of senior students.

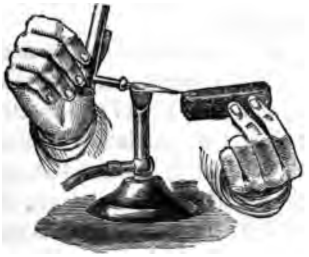
Two platinum wires should be kept in readiness, one to be used for borax beads and the other for flame colorations, since a loop which has been used for borax colours the flame yellow.

33. *Ignition on charcoal in the blowpipe flame.*—Wood charcoal forms a very useful support for substances which are to be heated in the blowpipe flame. It cannot itself be melted, it conducts heat very badly, and possesses other properties which render it peculiarly adapted for ignitions. It is, if necessary, sawn into small pieces of the required shape and size with a fine-toothed saw.

Ignition on charcoal serves chiefly to detect the presence of metals; the substance for this purpose is heated either alone or mixed with certain other substances (usually sodium carbonate and potassium cyanide) in the inner blowpipe flame; globules or scales of the metal are thus frequently produced, also in many cases the oxide of the metal forms a characteristic coating or "incrustation" on the charcoal: an examination of the colour and properties of the metal or incrustation or of both, will frequently lead to the detection, or confirm the presence, of some metal. Charcoal is apt to leave a white ash when heated alone in the blowpipe flame; this must not be mistaken for an incrustation. A powder frequently requires to be moistened with water before being heated by the blowpipe flame, in order to prevent the flame from blowing it off the charcoal.

Exp. 50.—Choose a piece of wood charcoal free from large cracks, scoop out a small hollow near one end with a knife, and place in the hollow a small quantity of a mixture of powdered lead acetate and sodium carbonate or potassium-oxalate. Heat this mixture in the inner blowpipe flame, holding the charcoal in such a way that the rest of the flame plays over its surface (fig. 31). After heating for some time bright globules of lead will be seen in the cavity, and on removing the charcoal from the flame, the surface round the cavity will be found to be coloured with a yellow "incrustation."

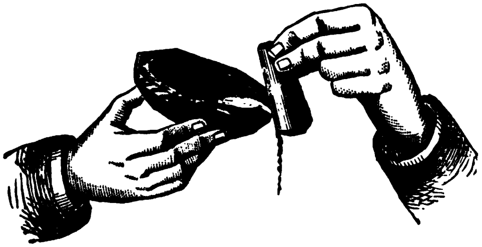
Fig. 31.



If one of the little globules of metal be then taken off the charcoal with the point of a penknife, and be struck smartly with a pestle on the bottom of a mortar turned upside down, it will flatten out into a cake, showing that the metal is "malleable" not "brittle." The metal lead is the only metal which gives a yellow incrustation and malleable globules; this test therefore serves to detect its presence.

33*a*. The melted mass left on the charcoal after fusing a metallic salt with sodium carbonate is best examined for metallic powder or scales by detaching it from the charcoal with the point of a penknife, then crushing it in an agate mortar or in a watch glass with a little water; on stirring and quickly decanting the water down a wet glass rod or pestle held against the edge of the vessel (fig. 32), the metallic powder, if present, will be left; by repeating this process of "levigation," the metal is obtained free from soluble substances and charcoal, it may then be further examined by a lens or magnet. A mixture of copper sulphate with sodium carbonate, when heated on charcoal and "levigated" as directed above, will furnish little red scales of copper: a mixture of ferrous sulphate and sodium carbonate will yield a grey powder consisting of metallic iron, which, when touched with the point of a magnetised knife, will become attached to it.

FIG. 32.



34. *Use of test-papers.*—Certain vegetable colours undergo remarkable changes when acted upon by chemical substances. Some of these are used for analytical purposes; the following experiment will explain how they are made use of.

EXP. 51.—Boil some pieces of a common red picklingcabbage leaf in water; a blue liquid will be produced. Pour some of this liquid into a test-tube and add several drops of hydrochloric acid, the colour will change to bright red; to another portion of the blue liquid add several drops of ammonia, the colour changes to green; to a third portion of the blue liquid add a little ammonium chloride solution, the colour remains unchanged.

Mix in a test-tube a little ammonia solution with three times as much distilled water, and add this weak ammonia gradually to the liquid reddened by several drops of hydrochloric acid; by careful addition the blue colour may be exactly restored, the acid previously added being "neutralised" by the ammonia.

Exp. 52.—Pour some blue litmus solution (made by boiling solid litmus in water and filtering) into three test-tubes; to the first add hydrochloric acid drop by drop until it turns distinctly red, preserve this liquid; to the second add ammonia solution, the liquid remains blue; to the third add ammonium chloride solution, the colour will not be affected.

Divide the reddened liquid into three parts; to one part add ammonia solution, the original blue colour is restored; to another part add hydrochloric acid, the red colour is unchanged; to the other part add ammonium chloride, the red colour remains unaltered.

These experiments may also be made with litmus test-papers, which are prepared by soaking paper in the blue and red liquids respectively, and allowing it to dry.

Exp. 53.—Repeat the preceding experiment, using the litmus papers instead of the solution, and moistening them with the liquid by dipping a glass rod into it and then touching the paper with the moistened end; the results mentioned in Exp. 52 will be obtained.

It will be seen then that the three substances employed differ in their action on litmus test-papers, as follows :—

Hydrochloric acid . . .	reddens blue litmus	does not affect red litmus
Ammonia	does not affect „ „	blues „ „
Ammonium chloride . .	„ „ „ „ „	does not affect „ „

Each of these substances is a sample of a whole class, the members of which resemble it in their action on litmus.

The class of *acid substances*, including sulphuric acid, nitric acid, &c., and many metallic salts, resemble hydrochloric acid in reddening blue litmus and not affecting red.

The class of *alkaline substances*, including potassium hydrate, sodium hydrate, &c., and many salts of the alkalis, resemble ammonia in blueing red litmus and not affecting blue.

The class of *neutral substances*, including potassium chloride, sodium chloride, &c., resemble ammonium chloride in not affecting either blue or red litmus.

Hence in analysis we use red and blue litmus-papers to test whether a liquid is "acid," "alkaline," or "neutral."

The red cabbage liquid, as will be seen, is better suited for a test-paper than litmus, since one paper shows both alkalis and acids; but the colour is very apt to spoil with keeping.

Sometimes a liquid in which a coloured precipitate is suspended has to be tested by litmus-paper, the precipitate then frequently hides the colour of the paper when a drop of the liquid is placed upon it. In such a case moisten the paper with a drop of liquid, and wash the liquid off after a short time with a stream of distilled water from the wash-bottle; the colour of the paper is then distinctly seen.

35. *Making neutral, acid, and alkaline.*—An acid substance by being mixed with a proper proportion of an alkaline substance becomes "neutral;" similarly an alkaline substance is rendered "neutral" by mixture with a proper quantity of an acid. Thus by mixing hydrochloric acid and ammonia in the proper proportions, the neutral substance ammonium chloride is formed; the acid is then said to be "neutralised" by the alkali, or the alkali by the acid.

It is often required to "neutralise" an acid with an alkali or *vice versâ;* the following experiment will show how this process of neutralising an acid is performed.

Exp. 54.—Pour a little hydrochloric acid into a porcelain dish, add to it ammonia several drops at a time, keeping the liquid constantly stirred with a glass rod, and occasionally touching a slip of blue litmus-paper with the end of the rod moistened by the well-stirred liquid; as soon as the paper is only faintly reddened by the liquid, the ammonia is added very carefully, stirring well after the addition of each drop, and trying the action of the liquid by touching the wet glass rod against first blue and then red litmus-paper, waiting a short time to give the liquid time to act on the paper. When the colours of both blue and red paper remain unaffected, the process of "neutralisation" is completed.

It is well to add *dilute* ammonia towards the end of the process; this is prepared by mixing a little ammonia with three or four times as much distilled water in a test-tube.

The process of neutralising may often be simplified when the liquid is clear and colourless, by dropping into it a small piece of blue litmus-paper, the colour of which is noted after each addition of ammonia; the condition of the liquid should, however, even in this case be proved towards the end by moistening with it both red and blue paper.

The process of neutralising an alkali with an acid is precisely similar to that of neutralising an acid with an alkali, except that red litmus is at first used instead of blue.

35 *a.* It will be readily understood from what has been said above, that if an alkaline or neutral liquid is to be "acidified," acid is added gradually as was described in the preceding experiment, until a drop of the well-mixed liquid reddens blue litmus paper. If an acid or neutral liquid has to be made alkaline, the alkali is added until a drop of the liquid turns red litmus blue. These processes are often termed adding acid or alkali "in excess."

35 *b. Yellow turmeric paper* is occasionally employed instead of red litmus to test for alkalis, which turn it reddish-brown; it is unaffected by acids, with the exception of boracic acid. The most important use for turmeric paper is to test for boracic acid, the solution of which, if dried on the paper at a gentle heat, turns it orange-red.

Exp. 55.—This may be shown by stirring a little borax upon a watch-glass with dilute hydrochloric acid until it is dissolved, then moistening the lower part of a slip of turmeric paper with this liquid, and holding it some distance above the flame, or better, placing it in a steam oven until dry; the portion of the slip which was moistened will now appear reddish-brown, the colour being well seen by contrast with the light yellow upper portion of the paper.

FRACTIONAL SOLUTION, CRYSTALLISATION, AND DISTILLATION.

1. *Fractional solution.*—Since different substances show differences in solubility in water and acids, it is often possible by means of solvents to separate the constituents of a mixture. This process is termed separation by "fractional solution." Illustrate this by making a mixture of powdered ferrous sulphate ($FeSO_4.7H_2O$) and ferric oxide (Fe_2O_3); on warming a portion of this with water, $FeSO_4.7H_2O$ alone dissolves, leaving Fe_2O_3: if another portion is warmed with HCl, both substances dissolve.

2. *Fractional crystallisation.*—If a solution of two different salts be boiled down in an evaporating basin, one salt will usually furnish crystals before the other does, and by this process of "fractional crystallisation" a separation of the salts may often be effected. Boil down a solution of sodium chloride (NaCl) and nitre (KNO_3): NaCl will crystallise out of the boiling liquid in hollow cubes, and on cooling prisms of nitre will separate.

3. *Fractional distillation.*—Liquids which boil at different temperatures may often be separated by distillation (p. 24); that part of the mixture which first distils over consisting chiefly of the liquid with the lowest boiling-point, and the successive portions of the distillate becoming richer in the liquids of higher boiling-points. The process of "fractional distillation" may be illustrated by distilling a mixture of alcohol and water; since alcohol boils at a lower temperature than water, the first part of the distillate contains the greater part of the alcohol, as is shown by its burning and by its dissolving camphor or iodine in quantity. The last portion of the distillate does not give these tests for alcohol, and is proved to be water by turning dry $CuSO_4$ blue and dissolving it.

SECTION IV.

ANALYTICAL REACTIONS.

Course of Practical Analysis to be pursued by the Student.—The text is so arranged that two main courses may be framed from it in this and the following sections. The one is suited to senior students, and dispenses with the training in the detection of simple substances, which, although useful as a stepping-stone to the full analytical method, is in itself of only small importance. The other course is suited to junior students, and forms an easy approach to the more difficult complete methods of analysis; it teaches the plan of analysing simple substances which may contain one acid-radicle and one metallic-radicle. This course is therefore also suited to those who are learning analytical chemistry with the view of preparing themselves for examinations in which the candidate is not required to detect more than one acid-radicle and one metallic-radicle. These courses are described below as the "Senior course" and "Junior course."

The Senior course consists in working through the analytical reactions in Section IV.; as those for each group are completed, several substances whose composition is unknown to the student are tested for one member only by the "Table of differences" at the end of the group: as soon as these are detected with readiness, substances which may contain two or more of the members mixed together are analysed by the Group-table in Section VI.: a reference to the "Table of differences" will show at a glance on what principles these Group-tables are founded, and this is further explained by a statement following the "Table of differences." This plan of working with each Group-table before going on to the reactions for the next group is to be recommended, since, whilst the differences upon which the separation and special tests of the elements in the groups are fresh in the student's memory, they are practically applied. The Analytical tables are thus gradually rendered intelligible one after another, whilst the student is proceeding through the reactions. The book is so arranged, however, that the Analytical tables may be left until the reactions for all the groups have been finished. When all the groups have been worked through in this manner, the analysis of substances is commenced which may contain any members of any of the different analytical groups; the directions for the systematic method of analysis required for this purpose being contained in Section VI.

The Junior course.—Since this is intended to teach only the detection of one metallic- and one acid-radicle, the student, whilst working through Section IV., omits all those paragraphs marked "s" after the number, relating to the separation and detection of the mixed members of a group; and after finishing the reactions for each group, he proceeds to detect the members occurring singly in several substances of unknown composition by the Table of differences and by reference to the reactions. After working in this way through Section IV., he at once proceeds to analyse simple substances by Section V.

As stated above, this Junior course may occasionally be made to precede the Senior with advantage—that is to say, the student, after working through the Junior course and learning how to detect simple substances, may analyse mixtures containing two or more members of each group by the directions given at the end of each of the groups in Section IV., and may then proceed to the full method of analysis contained in Section VI.

INTRODUCTORY REMARKS ON ANALYTICAL CHEMISTRY.

The science of chemistry reveals to us the fact that every substance on this earth, and probably in the universe, consists either of one kind of matter only, or of two or more different kinds which we cannot further separate. Those substances which contain only one kind of matter are called "chemical elements"; their number at present amounts to a little over sixty. A list of them is given in paragraph 558 at the end of the book.

It is not often that these elements occur singly; two or more of them are commonly found together either in a state of mere "mixture," or united in a much more intimate manner by the force of "chemical affinity" to form a "chemical compound."

Analytical chemistry or analysis (literally meaning an *unloosing* or *separation*) teaches us how to discover what element, elements, or sometimes what groups of elements, any substance of unknown composition contains: it also enables us to ascertain whether any particular element or group of elements is present in a given substance. In order to become a thorough analyst, it is necessary to be intimately acquainted with the properties of every element and of the compounds it forms with other elements, and therefore efficiency in analysis depends in a large measure upon the knowledge of what is usually termed "theoretical," or more appropriately perhaps "descriptive chemistry." But although we recognise a substance by its properties, still a knowledge of *all* its properties is not requisite to enable us to detect its presence: for ordinary analytical purposes certain marked properties of each element or group of elements are selected, and by these properties called "tests" or "reactions" we detect its presence.

The "tests" or "reactions" most frequently selected are the following:—1st, The behaviour of a substance when heated under various conditions; and 2d, Its behaviour when mixed with certain liquid or solid substances: such substances, added for analytical purposes, are called *reagents.* Some reagents serve for detecting one element or compound only, and are therefore called *special reagents;* others separate or detect a group of elements or compounds, and are called *group reagents* or *general reagents:* these latter reagents serve to separate substances into "Analytical groups," as will be more fully explained hereafter.

The student must bear in mind that, although an acquaintance with the most important reactions of substances is indispensable to success as an analyst, caution, skill, and neatness in manipulation are not less necessary, and the most scrupulous attention must be paid to all directions bearing on these matters.

36. *Analytical classification.*—The chemical elements are frequently divided into two classes—viz., *metals* and *non-metals.* These classes form, with a slight modification, convenient divisions for analytical purposes. In the class of "metals" must be included the hypothetical metallic-radicle ammonium (NH_4), also the element hydrogen. The class of "non-metals," either singly, or in groups formed by their combination *inter se* or less frequently with a metal, unite with hydrogen and with metals forming "salts"; the hydrogen salts of these radicals are usually called "acids." Thus Cl*, SO_4, PO_4, MnO_4 form the hydrogen salts or acids, HCl, H_2SO_4, H_3PO_4, $HMnO_4$ and the metallic salts, KCl, $MgSO_4$, Na_3PO_4, $KMnO_4$: metal-hydrogen-salts or acid-salts, such as $NaHSO_4$, Na_2HPO_4, are also known. The general name "acid radicle" is conveniently applied to Cl, SO_4, PO_4, MnO_4, &c., it being understood that although most of these are unisolated compound radicles,—*i.e.*, groups containing more than one element—some of them are elements; their common property is that of forming in combination with hydrogen and with metals—salts. For analytical purposes, then, we may separate the substances we have to detect into the groups of Metals and Acid radicles. In trying the reactions for these substances, it is usual to employ, not the substances themselves, but certain compounds—usually salts—which contain them.

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* The meaning and use of chemical symbols will be briefly explained hereafter (40); but the student is presumed to be already acquainted with them. In par. 558 will be found a list of the symbols of the different elements.

For convenience in hasty writing, it is not uncommon to employ abbreviations for the compound radicles, especially for such as are constantly met with or are of unusual complexity. In the text the only such abbreviations employed are:—

Am for (NH_4), the metallic radicle ammonium.
$\overline{\mathrm{T}}$,, $(C_4H_4O_6)$, the acid radicle of tartaric acid and the tartrates.
$\overline{\mathrm{A}}$,, $(C_2H_3O_2)$, the acid radicle of acetic acid and the acetates.
$\overline{\mathrm{Cfy}}$,, $(FeCy_6)$, the acid radicle of ferro- and ferri-cyanides.
Ho ,, (HO), the radicle hydroxyl, occurring in hydrates, &c.

37. *Analytical groups.*—The metals are separated by their behaviour with certain general reagents into five principal groups, two of these being furthur subdivided into two subgroups. Each of these groups receives a distinctive name—either a number from its place in the system, or a name from some conspicuous or important member of the group, or from the general reagent used to precipitate the group. Thus we speak indifferently of the 4th Group, the Barium Group, or the Ammonium-carbonate Group. On pages 168 and 169 will be found a table showing the analytical groups with their distinctive numbers, names, and group reagents placed at the head of each column; the names and symbols of the elements contained in the group are arranged vertically beneath.

It is perhaps well to avoid referring to a group by its number, as different analysts number the groups differently, and the name thus given is therefore apt not to denote the group with certainty.

38. *Method of trying the Analytical reactions in Section IV.*—In order to become fully acquainted with the behaviour of different substances with "reagents" and thus to be able to detect them by their "reactions," each of these reactions should be performed with the substance itself or with one of its compounds, and the appearances presented carefully observed, so that the substance would be easily recognised in a body of unknown composition by means of those tests.

The following general rules must be carefully attended to in trying the reactions; they may be read through before commencing the reactions (par. 47):—

1. A *solution* of the substance which is directed to be used must always be employed, unless it is specially stated that the *solid* is required. Solid substances are required as a rule only for blowpipe reactions. A solution can be readily made from the solid, if necessary, by warming it with water or in some cases with dilute acid (529). This is often called the "original solution."

2. Commence by taking in a test-tube or small beaker a small quantity (about 15–20 c.c.) of the solution of the substance. If the solid substance is also required, take some of it upon a watch glass.

3. Pour a small portion of the solution into a perfectly clean test-tube, and add the first-mentioned reagent to it. The reagent must always be added in small quantity at first; more can be employed afterwards if excess is required. The student must from the first acquire the habit of working with small quantities both of solutions and reagents.

For each of the reactions a separate portion of the "original solution" is thus poured into a clean test-tube and the required reagent added to it.

4. Before trying each reaction the student must carefully read through the whole of the description of the results he is to obtain; he must then perform the experiment, literally following out the directions given. He must consider it a necessary condition of after success that each result is obtained precisely as stated in the text, and must never on any account pass on until he has conscientiously satisfied himself that the statements of the book are true, and that he could at any time repeat the test successfully.

39. *Entry in Note-book.*—Each reaction, as soon as it has been satisfactorily tried, should be neatly entered in the note-book in a short form. Since nothing so much aids brevity as the symbolic notation commonly used by chemists, the student should invariably adopt this chemical shorthand in entering results. A full account and explanation of chemical notation may be found in any work on theoretical chemistry, and merely a few hints will therefore be given here which will be of special use for the purpose of briefly entering reactions.

40. *Chemical notation.*—Each element has its corresponding *symbol* consisting of one or two letters; thus S stands for sulphur, Cl for chlorine. These symbols also stand for one *atom* of each element; by being written one after another they give the *formula* of a compound substance, showing what elements, and how many atoms of each of these elements, its molecule contains: thus KCl stands for one molecule of potassium chloride, a compound of one atom of potassium and one atom of chlorine; $PtCl_4$ stands for one molecule of platinic chloride, which consists of one atom of platinum combined with four atoms of chlorine, the small figure placed *below* a symbol to the right denoting how many atoms of that element are present. A number placed before a formula multiplies each symbol in that formula; thus $2PtCl_4$ signifies two molecules of platinum chloride, containing altogether two atoms of platinum and eight atoms of chlorine. If two or more symbols are included in brackets any number placed outside, either *before* or *below* the brackets, multiplies each symbol contained in the brackets; thus both $Sr(NO_3)_2$ and $Sr2(NO_3)$ stand for SrN_2O_6, and this may also be written $Sr2NO_3$. In paragraph 558 will be found a list of the elements, with their symbols and atomic weights; paragraphs 524, 525, 526, 529, and 530 contain a list of the names and corresponding formulæ of all the most common compound substances mentioned in the course of the book. Symbols and formulæ are always used in the text instead of names, but the student can readily find the corresponding names by reference to the above tables, or to the labels on the bottles which should contain both names and formulæ.

41. *Chemical equations.*—On adding a "reagent" to a substance some change usually occurs which gives rise to an alteration of colour, or very frequently to the formation of a precipitate having a certain colour, appearance, or behaviour with other liquids, which are characteristic of that particular substance, or of some element or group of elements contained in it; this change should be briefly described in the note-book in words, and then represented by a *chemical equation.* The rules for drawing out an equation may be found in any treatise on chemistry; a few general directions only are given here which will be of service for the special object in question.

42. *Rules for writing down an equation.*—Write down the formulæ of the two substances which are mixed together for the reaction, with the sign of addition (+) between them; then write the sign of equality (=), followed by the formula of the precipitate produced. In a complete equation the formulæ written on the left-hand side of the sign (=) are usually called the "left-hand side" of the equation; those to the right the "right-hand side." Since most of the following reactions are cases of "double decomposition"—*i.e.*, cases where a mutual exchange of certain elements or groups of elements occurs between the two compounds,—a little consideration will usually show how many molecules of the substances on the left-hand side of the equation are required to yield the formula for the precipitate, and also whether any other, and if so what other, substance is at the same time formed.

It is always necessary that the number of atoms of any one element on both sides of the equation should be equal. The following are dissected examples:—

The first test for K (47):—

$$KCl + PtCl_4 = \overline{K_2PtCl_6}.$$

This is simply placing together the substances mixed and the precipitate formed; the equation thus formed is evidently not correct, since we have two atoms of K on the right-hand side and only one on the left, also 6 atoms of Cl on the right and only 5 on the left; but this inequality is at once removed if we place 2 before the KCl, thus:—

$$2KCl + PtCl_4 = \overline{K_2PtCl_6}.$$

Evidently if the above equation is correct the precipitate is here the only substance formed, since the number of atoms in the substances on the left-hand side is exactly equal to that in the substance on the right.

Take now the first test for Ba (69):—

$$BaCl_2 + Am_2CO_3 = \overline{BaCO_3} +$$

This represents the substances mixed and the precipitate formed, but evidently the left-hand side of the equation contains more than the right (viz., Am_2 and Cl_2); hence we write it fully thus:—

$$BaCl_2 + Am_2CO_3 = \overline{BaCO_3} + 2AmCl,$$

which states that on mixing together barium chloride ($BaCl_2$) and ammonium carbonate (Am_2CO_3), we obtain barium carbonate ($BaCO_3$) and ammonium chloride (AmCl).

In writing out these equations a systematic method should be observed. Write down on the left-hand side of the equation first the substance whose reaction is being tried, then the reagent added; and on the right-hand side write first the formula for the precipitate with a line drawn over it by way of distinction, then any substance or substances which may be formed with it.

In the reactions which follow, the formulæ of the substance used, of the reagent added, and of the precipitate produced will always be given, these being sufficient data to enable the student to form the equation; only where the equation is exceptionally difficult or complicated will it be given in full.

43. The following contractions are recommended:—

expt.	for	experiment.	*insol.*	for	insoluble.
pp.	”	precipitate.	$sol^{n.}$	”	solution.
$pp^{n.}$	”	precipitation.	$cryst^{ne.}$	”	crystalline.
$pp^{d.}$	”	precipitated.	$col^{n.}$	”	coloration.
sol.	”	soluble.			

44. Examples are given below showing how the reactions should be entered in the note-book; they should be referred to as soon as the student commences the reactions for the metals:—

1st. [See pars. 47–50.]

POTASSIUM (K).—Used KCl.

$PtCl_4$: stirred on watch glass: yellow cryst$^{ne.}$ pp., sol. in much water and in KHO, insol. in alcohol:

$$2KCl + PtCl_4 = \overline{K_2PtCl_6}.$$

$NaH\bar{T}$: well shaken in a test-tube: white cryst$^{ne.}$ pp., sol. in water, KHO, and HCl:

$$KCl + NaH\bar{T} = \overline{KH\bar{T}} + NaCl.$$

Flame col$^{n.}$: pale violet, crimson-red through the indigo-prism or cobalt-glass.

Heated in tube or on platinum foil, solid KCl melted, but gave no fumes; hence not volatile.

2nd. [See pars. 69–74.]

BARIUM (Ba).—Used $BaCl_2$.

Am_2CO_3: white pp., sol. in $H\bar{A}$:

$$BaCl_2 + Am_2CO_3 = \overline{BaCO_3} + 2AmCl.$$

$CaSO_4$: heavy white pp., insol. in boiling HCl:

$$BaCl_2 + CaSO_4 = \overline{BaSO_4} + CaCl_2.$$

K_2CrO_4: light yellow pp., sol. in HCl, insol. in $H\bar{A}$:

$$BaCl_2 + K_2CrO_4 = \overline{BaCrO_4} + 2KCl.$$

Flame col$^{n.}$: on loop of Pt-wire, yellowish-green colour.

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45. *General rules to be observed whilst working.*—The student should attend carefully to the following precautions, which will most materially aid the progress and accuracy of his work; if they are observed from the commencement they will not be felt irksome:—

1. Before commencing work look through the reagent bottles belonging to the working bench (524); replace any which are absent, and arrange the bottles, if necessary, in the order indicated on an accessible list, or shown by the number on the labels of the bottles, or by the labels on the edge of the shelf. Then proceed to fill any which are empty and to re-label any whose labels are imperfect or loose. If any of the liquid reagents are not quite clear when shaken they must be filtered before use.

2. Arrange the apparatus required for use, on the hinder

part of the bench, so as to leave the front free to work upon; this will lessen the risk of upsetting anything.

3. Before commencing work, all glass and porcelain which is not in use should be carefully cleansed, if not already clean; but it is best never to put apparatus away dirty.

4. In using a reagent bottle take it in the right hand, remove the stopper by taking hold of it between any two of the fingers, or between the fourth finger and palm, of the left hand; hold the stopper in this way and replace it immediately after the bottle has been used. Pour the liquid out at the side opposite to that bearing the label, and prevent the last drop from running down the outside by touching the lip of the bottle either with the stopper or against the clean edge of the vessel. Solid reagents should be taken out of the bottle on a clean knife-blade or spatula, or with a spoon of wood or horn; or a small quantity should be turned out upon a piece of paper or upon the palm of the hand. *The bottle must be re-stoppered and replaced on the shelf in its proper place with the label outwards immediately after use, and must never be left standing on the working bench.*

5. If any solution or precipitate has to be put by for some time, always label it with a piece of gummed paper bearing an inscription or a reference number which is explained in the note-book and is sufficient to recall to mind what the solution or precipitate consists of: never trust to memory in this matter.

6. Brass crucible tongs must never be employed for holding hot evaporating basins containing acids, as some of the brass is apt to be dissolved and thus introduced into the solution. A hot dish may be moved either by holding it with a cloth or by lifting the stand upon which the dish is placed.

7. *Liquids only* are to be poured down the sinks; all *solid* refuse, such as soiled filters, broken glass, pieces of charcoal, &c., which would tend to stop the holes in the sink, must be thrown into a box placed in any accessible position in the laboratory, or into a small earthenware jar or vessel placed upon each working bench.

8. When a student is examining a substance to detect its composition, he must fully write down in his note-book each test or process, with the result obtained, as soon as it is completed. The analysis is thus written out gradually as it progresses: *on no account must the entry of the results be left until the completion of the analysis.* This rule cannot be too strongly enforced, as neglect of it is always attended with loss to the beginner. The most convenient form of entry is that of the Analytical Tables.

⁂ Cleanliness and neatness in analytical work cannot be too carefully attended to: more confusion and error may be caused by using dirty test-tubes, beakers, dishes, and funnels than would be thought possible by the beginner.

REACTIONS FOR THE METALS.

Note.—In trying through the reactions for the metals, the usual analytical order of the groups has been reversed, because the most simple reactions occur in the last groups, and they are therefore better suited for a beginner.

GROUP V.—POTASSIUM GROUP.

46. This group includes K, NH_4*, Na, Mg. The first three metals (K, NH_4, Na) are known as the "alkali metals."

The members of this group are not precipitated by any of the five group reagents.

Potassium (K).—Use KCl.

Refer if necessary to paragraphs 38 and 44 for directions how to work.

47. *$PtCl_4$* added after a drop of dilute HCl to some of the KCl solution on a watch glass and stirred with a glass rod, gives a yellow crystalline precipitate (K_2PtCl_6), forming first along the lines where the rod has rubbed the glass.

Stir up the precipitate and pour off the liquid with the precipitate into three test-tubes; to one add much distilled water and heat, the precipitate dissolves: to another part add alcohol, the precipitate does not dissolve. Hence this precipitate is soluble in water, insoluble in alcohol. By warming the third portion with solution of KHO, the precipitate is also shown to be soluble in that liquid.

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* A hypothetical metal radical, "Ammonium."

Remarks.—The following remarks are of great importance in using the above test for K. Since this precipitate is soluble in alkalis, the liquid should always be neutral or only faintly acid before adding $PtCl_4$; and since it is more insoluble in alcohol than in water, addition of alcohol causes the precipitate to form more rapidly in dilute solutions; owing to the solubility of the precipitate in water, very dilute solutions must however be evaporated nearly to dryness before applying the test. It is always well to add several drops of HCl before the $PtCl_4$, and to make sure by test-paper that the liquid is acid in reaction. These remarks apply also to the precipitate produced by $PtCl_4$ with ammonium salts (52).

48. *$H_2\overline{T}$ (or better $NaH\overline{T}$)* mixed with some of the solution in a test-tube gives a white crystalline precipitate ($KH\overline{T}$); usually this precipitate appears only when the mouth of the test-tube is closed with the thumb and the tube is well shaken for some time.

$$KCl + NaH\overline{T} = KH\overline{T} + NaCl.$$
$$KCl + H_2\overline{T} = KH\overline{T} + HCl.$$

Shake up and pour some of the liquid and precipitate into four test-tubes; add to these portions, water*, KHO, and HCl respectively; on being shaken and warmed the precipitate will dissolve in each of these liquids. Hence this precipitate is soluble in H_2O, KHO, and HCl. To the fourth portion add alcohol; the precipitate does not dissolve, since it is insoluble in alcohol.

Remarks.—In using the above test for the detection of K, the precipitate is not obtained at once in dilute solutions, but its formation is much hastened by the addition of alcohol: very dilute solutions must first be concentrated by evaporation. The solution to be tested should be neutral or only feebly alkaline: if acid in reaction, the acid should be neutralised by Na_2CO_3 or boiled off if volatile; feebly acid solutions may, however, be at once mixed with $NaH\bar{T}$; to an alkaline solution $H_2\bar{T}$ should be added till the reaction is strongly acid.

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* By "water" is always meant "distilled water," unless otherwise stated.

Note.—In the following reactions when a precipitate is stated to be soluble or insoluble in certain liquids, it must be proved to be so in the manner described in (47, 48) unless different directions are given.

49. *Flame coloration* (32).—Hold a loop of platinum wire in the flame and see that it gives no colour to the flame: then dip it into some KCl solution, or better still, moisten it and dip it into some powdered KCl, and again hold the loop in the upper part of the flame. A *pale violet* colour will be imparted to the flame if the KCl is pure*; but whatever colour is thus seen, the flame-colour will always appear *pale blue* or *violet* through the thinner portions of an indigo-prism, and deep *crimson red* through the thickest parts.

The light of a Bunsen flame itself usually appears pale blue through the indigo-prism: this colour should be noted before using the prism for examining a flame coloration.

The flame coloration due to potassium shows in the spectroscope a red line (α) and an indigo-blue line (β) (557).

Note.—A piece of blue cobalt glass may be used instead of the indigo-prism, but is by no means to be recommended, as the colour of different specimens is somewhat variable in shade and in intensity, whereas the colour of the indigo can be obtained of an invariable tint, by dissolving indigo-carmine in water.

50. Heat a little solid KCl in a small dry test-tube, or better on a piece of platinum foil; the KCl will melt but will not pass into vapour and produce white fumes unless heated very strongly, since K-salts are non-volatile at a moderate heat. The fused residue is often transparent and invisible if in small quantity; its presence is shown by a slight crackling being heard during cooling.

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* KNO_3 usually gives a purer potassium coloration.

AMMONIUM (NH_4, or Am).—Use NH_4Cl, (AmCl).

Note.—The symbol Am is often written for NH_4; it is very convenient, as it does away with the necessity of using brackets: *e.g.*, $Am_2S = (NH_4)_2S$.

51. *KHO** (*or NaHO*) poured into some of the solution, or upon the solid, in a test-tube and heated gives off NH_3 gas:

$$NH_4Cl + KHO = NH_3 + KCl + H_2O.$$

This gas is known by the following properties:—

a. Its pungent smell—that of common smelling salts.

b. By turning moistened red litmus-paper blue. A small piece of red litmus-paper is wetted with distilled water and placed upon a clean glass rod, and is then held in the test-tube, taking great care not to touch with it the liquid or the sides of the tube: the paper becomes blue.

c. A glass rod dipped into strong HCl or strong $\overline{H}A$, and held over the mouth of the test-tube, produces dense white fumes.

52. *$PtCl_4$* when stirred on a watch glass with some AmCl solution gives a yellow crystalline precipitate (Am_2PtCl_6); this precipitate is soluble in water and in KHO, but insoluble in alcohol. The remarks at the end of par. 47 apply here also. This precipitate if boiled with KHO evolves NH_3 (difference from K_2PtCl_6).

53. *$H_2\overline{T}$* (*or better $NaH\overline{T}$*) shaken for some time in a test-tube with AmCl solution gives a white crystalline precipitate ($AmH\overline{T}$), soluble in H_2O, KHO, and HCl, insoluble in alcohol. The remarks in par. 48 apply here also.

Note.—This precipitate is more soluble than the $KH\overline{T}$ precipitate, and therefore does not form so readily. Indeed with $H_2\overline{T}$ a precipitate rarely forms unless alcohol is added, in which the precipitate is less soluble than in water. With $NaH\overline{T}$ a precipitate is obtained on shaking the liquid well for some time unless it is very dilute.

54. *Flame coloration.*—AmCl, if pure, gives on platinum wire no colour to the flame.

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* In all cases where KHO is to be used NaHO may be substituted, and either KHO or NaHO may be used unless it is specially stated that "pure NaHO" is to be employed.

55. *Heat a little solid AmCl* in a small dry test-tube, or on a piece of platinum foil; it will be converted into vapour producing white fumes, which are seen if platinum foil is used by removing the foil for a moment from the flame: hence AmCl is volatile.

If the experiment is performed in a dry test-tube, a white coating or "sublimate" forms on the upper part of the tube.

SODIUM (Na).—Use NaCl.

56. *Flame coloration.*—NaCl gives an *intense yellow* colour to the flame; the colour is almost invisible or appears pale blue when viewed through the indigo-prism, *and never shows the slightest tinge of red or purple,* even when seen through the thickest parts of the prism. A crystal of red $K_2Cr_2O_7$ appears colourless if illuminated by this yellow flame. Examined by a small spectroscope, the sodium coloration gives a single yellow line (*a*) (557).

The yellow colour of the sodium flame is always readily seen, since it overpowers that of K; its production is the most reliable, and often the only possible, test for sodium.

Note.—If both K and Na are present, the K coloration is unseen and the yellow coloration of Na is alone visible; but on examining this flame through the indigo-prism or cobalt glass, the crimson coloration of the K is at once seen; prove this by mixing together KCl and NaCl and trying the flame coloration without and with the indigo-prism. The spectroscope also shows the lines of Na and K quite distinct from one another.

57. *Some solid NaCl heated* in a dry test-tube, or better on platinum foil, melts without producing white fumes unless heated very strongly, since NaCl is not volatile at a moderate heat.

MAGNESIUM (Mg.)—Use $MgSO_4.7H_2O$ solution.

58. *Na_2HPO_4** added after AmCl and AmHO gives a white crystalline precipitate ($MgAmPO_4$); if much water is present this precipitate forms only when the liquid is warmed and well stirred or shaken: it is soluble in acids (use HCl or HNO_3), insoluble in AmHO.

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* $NaAmHPO_4$ is better than Na_2HPO_4; since it produces the precipitate more readily than does Na_2HPO_4 in dilute solutions, and always in a crystalline condition.

$$MgSO_4 + AmCl + AmHO + Na_2HPO_4 = MgAmPO_4 + Na_2SO_4 + AmCl + H_2O.$$

59. *KHO* gives a white precipitate ($MgHo_2$)*, soluble in acids.

60. *AmHO* gives a white precipitate ($MgHo_2$); but if AmCl is added to the $MgSO_4$ solution before the AmHO, no precipitate is produced.

61. *Am_2CO_3* gives a white precipitate ($MgCO_3$), but only in strong solutions and on standing for some time: AmCl prevents the formation of this precipitate.

62. *Flame coloration.*—$MgSO_4.7H_2O$, if pure, gives no colour to the flame.

63. *Blowpipe test.*—A little solid $MgSO_4.7H_2O$ placed in a small cavity on a piece of wood-charcoal and then heated in the tip of the outer blowpipe flame, shines brightly and gives a white unmelted mass of MgO; no white fumes are given off since MgO is not volatile. If this white mass after cooling is moistened with several drops of cobalt-nitrate solution and again strongly heated for some time† in the outer blowpipe flame, it becomes *delicate pink* in colour: this colour is best seen when the mass is *quite cold*, by comparison with a white piece of paper held near the residue on the charcoal.

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* This convenient symbol (Ho) for the group hydroxyl, first proposed by Frankland, will be employed throughout in the formulæ for all hydrates containing more than one atom of hydroxyl; it prevents the necessity of using brackets, as a number placed below the symbol multiplies both H and O, thus Ho_2 equals $(HO)_2$ or H_2O_2; and Ho_3 equals $(HO)_3$ or H_3O_3.

† It is necessary to heat strongly after moistening with $Co(NO_3)_2$, else blue $Co(NO_3)_2$ remains.

64. Group V.—Table of Differences.

Note.—A blank (thus ——) signifies that no marked or characteristic change occurs. For the method of using the table see par. 65.

Tests.	K—salts.	NH$_4$—salts.	Na—salts.	Mg—salts.
* *a. For solutions.*				
1. Boil with KHO solution:	——	NH$_3$ gas given off: known by its smell and by turning moist red litmus blue; it also gives dense white fumes with strong HCl.	——	White precipitate.
2. Heat in flame on clean platinum wire:	*Pale violet flame*, which appears crimson through the indigo-prism.	——	*Intense yellow* flame, which shows no tinge of red through the indigo prism.	——
3. Add AmCl, AmHo and Na$_2$HPO$_4$:	——	——	——	White crystalline precipitate.
4. Add PtCl$_4$ and a drop of dilute HCl, and stir vigorously:	Yellow crystalline precipitate, forms only in strong solutions.	Yellow crystalline precipitate, forms only in strong solutions.	——	——
5. Add $H_2\bar{T}$ (or better $NaH\bar{T}$) and shake well:	White crystalline precipitate in strong solutions.	White crystalline precipitate in strong solutions.	——	——
b. For solids only.				
6. Heat to redness in a dry test-tube, or on platinum foil:	Non-volatile, unless heated to bright redness.	Volatile; white fumes are given off and the substance passes into vapour (at least partially) forming a sublimate on the upper part of the tube.	Non-volatile, unless heated to bright redness.	Non-volatile.
7. Heat strongly on charcoal in the outer blowpipe flame:	Melt easily and sink into the charcoal, colouring the flame pale violet. (See 2.)	Are volatile, giving off white fumes.	Melt easily and sink into the charcoal, colouring the flame intense yellow. (See 2.)	Leave ultimately a white infusible residue which shines brightly, and which if moistened with Co(NO$_3$)$_2$ solution and reheated becomes pink.

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* In using these tests for the detection of a single member of the group in solution, only the first four need be employed; the fifth test may be substituted for the fourth. Tests 6 and 7 are used only for the examination of solids. They may be supplemented, however, by tests 1 and 2.

65. The method of using the foregoing table may be explained in a few words. Suppose a *solution* to be given which is known to contain one member of this group, the first five tests in the table (four and five being alternative) will enable us to decide which member is present. It is simply necessary to examine *separate portions* of the solution by these tests, until a result is obtained which conclusively proves the presence of one of the four substances. Tests 1, 2, 6, and 7 are similarly employed for examination of a *solid* substance. The student should never rest content with one test only when a second can be tried in confirmation, and he should always consider the tables of differences as mere summaries, and should refer back to the separate reactions for fuller accounts of the tests when required.

The results of these analyses, and of all analyses subsequently made by using the tables of differences, are best entered in three parallel columns, as shown in the upper part of the table in par. 67. The statement of the experiment or test performed is entered in the first column, the result observed being placed beside it in the second column, whilst beside this in the third column is stated the conclusion inferred from the result obtained.

At the end of each analysis state the result thus:—

Found K.

66. (s)* *Detection of Members mixed.*—We can also devise from the above table a plan for detecting the members of this group when mixed together. It is evident—

1. That NH_4 can always be detected by boiling with KHO solution.

2. That Na can always be detected by its yellow flame coloration.

3. That Mg can always be detected by giving a white precipitate on addition of AmCl, AmHO, and Na_2HPO_4 to its solution.

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* Paragraphs marked with an (s) are intended for a student using the senior course, and are passed over in the junior course.

4. That K may always be detected by giving a flame coloration which appears crimson through the indigo-prism.

5. That the confirmatory test for K, by stirring with $PtCl_4$, may be tried if no NH_4 is present; but it cannot be employed when NH_4 has been found, since NH_4 gives a yellow precipitate exactly like that given by K. Hence NH_4 is first removed by evaporating some of the solution to dryness, and strongly heating the solid substance thus obtained on platinum foil as long as white fumes come off: the residue (if any) on the foil will then be free from NH_4, and can be dissolved and tested for K by $PtCl_4$.

The above remark concerning the $PtCl_4$ test applies also to the $H_2\overline{T}$ test for K, since NH_4 gives a precipitate with $H_2\overline{T}$ precisely similar to that yielded by K.

The following scheme which embodies the above tests, must be used to test for Mg, K, Na, and NH_4, when they may be present together.

The student should examine several substances by this table for the four members of Group V., writing down the results he obtains fully in the form adopted in the table; that is to say, three columns are ruled; in the first is described the test performed, in the second the result observed, in the third what substance is inferred from that result to be present or absent. After writing out the tests, the metals which have been found in the solution are stated, thus:—"Found K and Na."

⁂ Experiment IV. in the following Table may be omitted by a beginner, K being tested for by the flame coloration alone (Exp. II.)

67. (s.) TABLE FOR TESTING FOR Mg, K, Na, NH_4, IN A SOLUTION WHICH MAY CONTAIN ONE OR ALL OF THEM.

(See note at the top of page 83.)

Experiment.	Observation.	Inference.
I. Add to a small part of the solution AmCl, then AmHO and Na_2HPO_4, shake well and allow to stand if no precipitate appears at once.	1. A white precipitate forms.	Presence of Mg.
	[2. No precipitate appears.]	[Absence of Mg.]
II. Dip a small loop of clean platinum wire into the solution, and hold it near the top of the Bunsen flame.	1. A yellow flame.*	Presence of Na, possibly also of K.
	Look at the yellow flame through the indigo-prism; a crimson red colour is seen.	Presence of K.
	2. A pale violet flame, appearing crimson red when seen through the indigo-prism.	Presence of K and absence of Na.
	[3. No colour is imparted to the flame.]	[Absence of K and Na.]
III. Boil some of the solution in a test-tube with KHO solution, and try whether NH_3 is given off by smelling; or if it cannot be smelt, by testing with moistened red litmus-paper or strong HCl.	1. NH_3 gas is smelt, or the red litmus becomes blue, and white fumes are obtained with HCl.	Presence of NH_4.
	[2. No NH_3 is smelt, and moist red litmus paper is not blued, neither are white fumes obtained with HCl.]	[Absence of NH_4.]

IV. A confirmatory test for K may be made by adding $PtCl_4$ or $NaH\overline{T}$. If Exp. III. has shown that NH_4 is present, proceed to test for K by column A; if NH_4 is absent, test for K by column B (66, 5).

A.	B.
Boil some more of the solution down to dryness in a porcelain dish; scrape out the white substance left in the dish, and heat it strongly on a piece of platinum-foil as long as any white fumes are seen to be given off when the foil is taken out of the flame for an instant. Place the foil when cold in a test-tube, and boil it with *very little* water, to which a drop of dilute HCl has been added, and evaporate to several drops; cool the liquid, and pour it out upon a watch glass, add a little $PtCl_4$†, and stir well:— A yellow precipitate:—*Presence of K.*	Pour a little of the solution upon a watch glass, add $PtCl_4$, and stir well: a yellow precipitate:—*Presence of K.* If no precipitate forms, K may nevertheless be present, the solution being too dilute to allow the precipitate to form; therefore pour some of the solution into a porcelain dish, boil down nearly to dryness, cool and test this liquid by stirring it with $PtCl_4$† on a watch glass, adding alcohol if the precipitate does not appear at once: a yellow precipitate:—*Presence of K.* *Note.*—Addition of alcohol will frequently produce the precipitate without evaporation.

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* Almost all substances contain traces of Na, and therefore when heated in the Bunsen flame tinge it more or less yellow; hence, unless the yellow coloration is very intense, enter in the results "No trace."

† In this and all subsequent places where $PtCl_4$ is directed to be used as a test for K, the $NaH\overline{T}$ test may be substituted for economical reasons.

Note.—Separate portions of the solution are to be used for each of these experiments. It must be understood that *only one* of the numbered results in the second column can be obtained in each experiment, but one or other of these must invariably be observed. In subsequent tables, that result only is stated in the second column which indicates the presence of the substance, and the absence of that result proves the absence of the substance: the portions in brackets will therefore be omitted in future.

GROUP IV.—BARIUM GROUP.

68. This group includes Ba, Sr, Ca.

The members of this group differ from those of Group V. by being precipitated as carbonates by Am_2CO_3, even in the presence of AmCl[*]. They are not precipitated by any other of the group reagents.

BARIUM (Ba).—Use $BaCl_2.2H_2O$ [†].

69. *Am_2CO_3* added after some AmCl: white precipitate ($BaCO_3$) which is at first flocculent, but if heated and allowed to stand, slowly shrinks in bulk and becomes crystalline.

Pour a little of this into another test-tube and add to it some $H\bar{A}$, the precipitate dissolves entirely if sufficient $H\bar{A}$ is added.

70. *$CaSO_4$ or H_2SO_4:* heavy white precipitate ($BaSO_4$), insoluble in HCl even on boiling.

71. *K_2CrO_4* added after some $H\bar{A}$: yellow precipitate ($BaCrO_4$), soluble in warm HCl, insoluble in warm $H\bar{A}$. (Diff. from Sr.).

[‡] **72.** *H_2SiF_6:* semi-transparent precipitate ($BaSiF_6$): the precipitate often does not appear in dilute solution until after some time, or until the liquid is boiled, or alcohol is added. Shaking or stirring the liquid hastens its formation.

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[*] On *boiling* with AmCl solution, however, the precipitates are more or less perfectly dissolved.

[†] These two molecules of water are termed "water of crystallisation," and are always present in *crystallised* barium chloride.

[‡] A reaction in small type is comparatively unimportant, and may be read through and omitted by beginners.

73. *$Am_2C_2O_4$:* white precipitate (BaC_2O_4).

74. *Flame coloration:* $BaCl_2$ gives a yellowish-green colour to the flame, visible through the indigo-prism.

The spectrum consists of a number of lines, the most characteristic of which are three green lines, α, β, γ. (557).

STRONTIUM (Sr). Use $Sr(NO_3)_2.4H_2O$.

75. *Am_2CO_3* added after some AmCl: white precipitate ($SrCO_3$), soluble in $H\bar{A}$. This precipitate is flocculent at first, but if heated it quickly becomes crystalline and shrinks very considerably.

75 *a. $CaSO_4$ or H_2SO_4:* white precipitate ($SrSO_4$), this precipitate does not usually form at once in a cold solution, but only after standing for some time. The precipitate, however, *appears at once on boiling the liquid.*

76. *K_2CrO_4* added after some $H\bar{A}$: no precipitate, since $SrCrO_4$ is soluble in $H\bar{A}$.

77. *H_2SiF_6:* no precipitate.

78. *$Am_2C_2O_4$:* white precipitate ($SrCr_2O_4$).

79. *Flame coloration*: crimson-red; this colour appears *intense red* through the indigo-prism unless the flame-coloration is very faint.

The strontium spectrum contains many lines; the most characteristic are the orange line (α), the red lines (β, γ), and the blue line (δ) (557.)

CALCIUM (Ca).—Use $CaCl_2.6H_2O$.

80. *Am_2CO_3* added after AmCl: white precipitate ($CaCO_3$), soluble in $H\bar{A}$. This precipitate is flocculent at first, but on standing for some time or on being gently heated, it shrinks considerably and becomes crystalline.

81. *$CaSO_4$:* no precipitate, even on standing or on being boiled.

82. *H_2SO_4:* white precipitate ($CaSO_4$) forms at once in strong solutions, and often in weak solutions on being boiled; but since $CaSO_4$ is not quite insoluble in water, some of it will always remain dissolved; prove this by boiling the liquid containing the precipitate and filtering it, then keep adding AmHO to the filtrate and stirring it until a drop of the solution turns red litmus blue and the liquid smells of NH_3, then add $Am_2C_2O_4$, a white precipitate will form, showing the presence of Ca (85) which must have been dissolved as $CaSO_4$.

83. K_2CrO_4: no precipitate.

84. H_2SiF_6: no precipitate.

85. $Am_2C_2O_4$: white precipitate (CaC_2O_4), soluble in most acids except $H\underline{A}$ and $H_2C_2O_4$.

86. *Flame coloration*: yellowish-red; this colour viewed through the indigo-prism *does not appear red* but *dingy green*, thus differing from that given by Sr.

In the calcium spectrum the most characteristic lines are the green line (β), and the intense orange line (α) (557).

87. Group IV.—Table of Differences.

The first three tests only need be used for the detection of a member of this group occurring by itself in solution : test 1 serves also for solids.

Tests.	Ba—salts.	Sr—salts.	Ca—salts.
1. Flame coloration:	Greenish-yellow.	Intense crimson, appears *red* through the indigo-prism.	Yellowish red, appears *dingy green* through the indigo-prism.
2. $CaSO_4$ added to the cold solution:	An immediate precipitate.	A precipitate appearing only after a short time *or immediately on boiling*.	——
3. K_2CrO_4 added after $H\underline{A}$:	Light yellow precipitate.	——	——
[4. H_2SiF_6 added:	White precipitate.	——	——]
5. H_2SO_4 added in excess and boiled:	*Entirely* precipitated as $BaSO_4$.	*Entirely* precipitated as $SrSO_4$.	*Partially* precipitated as $CaSO_4$; hence the filtrate from the precipitate contains Ca, which may be detected by adding AmHO in excess, and then $Am_2C_2O_4$; a white precipitate forms.

When analysing a solution which is known to contain only one member of this group, its detection by the first three of the above tests will be exceedingly simple.

88. (s) *The separation and detection of Ba, Sr, and Ca, when mixed,* depends upon the following differences :—

1. The flame colorations, examined if necessary through the indigo-prism, or by means of the spectroscope.

2. The difference in behaviour with $CaSO_4$, which at once indicates the presence or absence of Ba.

3. The separation of Ba, if present, by K_2CrO_4 in the presence of $H\bar{A}$.

4. The precipitation of Sr by $CaSO_4$ on being boiled, which will indicate the presence of Sr in the absence, or after the separation, of Ba.

5. The precipitation of Sr, if present, by boiling with H_2SO_4 ; sufficient $CaSO_4$ then remaining in the solution to give, after making the filtrate alkaline with AmHO, a precipitate with $Am_2C_2O_4$.

89. (s) A solution which has to be examined for Ba, Sr, and Ca, and can contain only these substances, is made alkaline, if not already so, by addition of AmHO ; then Am_2CO_3 is added as long as it is seen to cause any precipitate after the liquid has been stirred well and allowed to settle. The liquid is then filtered, and a little more Am_2CO_3 added to the clear filtrate ; if any further precipitate forms, more Am_2CO_3 is added and the liquid is again poured through the same filter, pouring the first portion of the filtrate once more through the filter as it is sure to come through turbid. As soon as the filtrate gives no further precipitate with Am_2CO_3, all the members of this group which were present in the solution have been precipitated as carbonates, and on filtration are obtained on the filter. The precipitate is then examined by Table IV. (438).

Note.—In using the group Tables in Section VI. it must be understood that they are drawn out to meet the case of all the members of the group being present. If therefore in any case no precipitate forms on addition of a reagent, the substance or substances whose presence would have been indicated by the formation of the precipitate must be absent. Hence a filtration which is directed to be made is often unnecessary, *and the solution* itself is treated as is directed for the filtrate.

GROUP III.A.—IRON GROUP.

90. This group includes Al, Fe, Cr.

The members of this group differ from those of Groups III.B, IV, and V., by being precipitated by AmHO after addition of AmCl; they are not precipitated however by the group-reagents for Groups II. and I.

This group is also precipitated by Am_2S, or by H_2S added to the alkaline solution.

Group III.A. further differs from Group III.B. in being completely precipitated by adding $BaCO_3$ shaken up with water; this reagent affords the most perfect means of separating the two groups.

The members of this group show no characteristic flame colorations; but with the exception of Al, they give borax beads of characteristic colour.

ALUMINIUM (Al).—Use $AlAm(SO_4)_2.12H_2O$ ammonia-alum, or $AlK(SO_4)_2.12H_2O$ potash-alum.

91. *AmHO:* white flocculent precipitate (Al_2Ho_6), which is seen best on boiling the liquid: soluble in HCl and in $H\bar{A}$; slightly soluble in AmHO, especially in the absence of AmCl:—

$$2AlAm(SO_4)_2 + 6AmHO = Al_2Ho_6 + 4Am_2SO_4 .$$

92. *KHO* added in very small quantity: same precipitate as AmHO: soluble in excess of KHO. If to a part of the KHO solution HCl be added gradually until it is neutral, the Al_2Ho_6 is reprecipitated; if more acid is added the precipitate dissolves, but is reprecipitated by adding AmHO in excess. From another portion of the KHO solution the Al_2Ho_6 may be reprecipitated by addition of sufficient AmCl.

93. *Am_2S:* same precipitate (Al_2Ho_6), H_2S gas coming off or remaining dissolved in the liquid:—

$$2AlAm(SO_4)_2 + 3Am_2S + 6H_2O = Al_2Ho_6 + 4Am_2SO_4 + 3H_2S.$$

94. *Blowpipe reaction.*—Some solid $AlAm(SO_4)_2.12H_2O$ heated on charcoal in the outer blowpipe flame, then moistened, after cooling, with $Co(NO_3)_2$ solution and again heated in the outer flame, gives a *fine blue mass.*

95. IRON (Fe).—Two classes of iron compounds are known, which differ in appearance and properties and behave differently with reagents; they are distinguished as *ferrous* and *ferric* compounds respectively, the former being supposed to contain the metal *ferrosum* (Fe'')*, the latter *ferricum* (Fe'''). It is usual in stating analytical results to mention in which state of combination the iron exists, and therefore some distinctive tests are described below (101). The whole of the reactions of ferrous compounds are not given, because Fe, if present, is always separated and detected in the course of analysis as ferricum, and ferrosum is tested for by special reactions in the original substance. Ferrous- are readily converted into ferric-compounds by boiling for a short time with a little strong HNO_3, or with HCl and a crystal of $KClO_3$.

FERRICUM (Fe''').—Use Fe_2Cl_6.

Ferric salts are usually yellow or reddish-yellow; ferrous salts usually pale green, or white if perfectly dry.

96. *AmHO* or *KHO:* reddish-brown, flocculent precipitate (Fe_2Ho_6): insoluble in KHO, soluble in HCl.

97. *Am_2S:* a black precipitate (FeS), mixed with white S: soluble in boiling acids, insoluble in KHO :—

$$Fe_2Cl_6 + 3Am_2S = \overline{2FeS + S} + 6AmCl$$

In a very dilute solution of Fe a green colour is produced at first by Am_2S, and the black precipitate separates only after a time.

98. *H_2S*†*:* same black precipitate in alkaline solutions: in neutral or acid solutions S is precipitated and the ferric is changed to a ferrous salt, the colour of the solution changing from yellow to pale-green as is seen after boiling and filtering:

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* This distinction of the two conditions of Fe and other elements by dashes or roman numbers placed above the symbol, first proposed by Odling, is very convenient for brevity.

† H_2S may be added as a solution of the gas in trying the reactions; *in the course of* analysis the gas is usually passed into the liquid.

$$Fe_2Cl_6 + H_2S = 2FeCl_2 + 2HCl + S.$$

99. *Blowpipe reactions.*—These are the same for ferrous as for ferric compounds. Fused with Na_2CO_3 on charcoal in the inner flame a grey magnetic powder is left (33 *a*); this is shown to be magnetic by being attracted when touched under water by the end of a magnet, or of a magnetized knife-blade.

100. *Borax bead:* Outer flame: *reddish-brown* while hot: *yellow* when cold.
Inner „ *greenish* bead, the colour of green bottle glass.

101. DISTINCTIVE TESTS FOR FERROUS AND FERRIC SALTS.

For these tests several drops of dilute HCl should be first added to the Fe solution.

Reagents to be added.	Ferric-salts. (Use Fe_2Cl_6 solution.)	Ferrous-salts. (Use $FeSO_4$ solution.)
1. *K_4FeCy_6* added in small quantity:	*Dark-blue precipitate,* "Prussian blue"; insoluble in HCl*, soluble in $H_2C_2O_4$, and slightly soluble in K_4FeCy_6 added in excess; turned brown by KHO.	*Light-blue precipitate,* becoming dark-blue in the air, or on addition of HNO_3 or Br-water.
2. *$K_6(FeCy_6)_2$*:	*No precipitate:* the solution darkens, but on dilution with water is seen to contain no precipitate.	*Dark-blue precipitate,* "Turnbull's blue": insoluble in HCl. In *very* dilute solution only a dark bluish grey colour is produced.
3. *KCyS*:	*Blood-red coloration:* no precipitate is produced, the liquid being perfectly clear on dilution; the colour is immediately destroyed when several drops are poured into some $HgCl_2$ solution: its production is hindered by HNO_3 and by $\bar{H}A$.	*No red coloration* unless small quantities of ferric-salts are present.
4. *KHO*:	*Reddish-brown* precipitate.	*Dingy-green* precipitate, turning brown in the air.

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* The solubility and insolubility of this precipitate must be shown by warming separate portions of the blue liquid containing the precipitate with HCl, $H_2C_2O_4$ and K_4FeCy_6, then separately filtering each: if the precipitate has been dissolved the filtrate will be dark blue.

Chromium (Cr).—Use $CrK(SO_4)_2.12H_2O$, chrome-alum.

Chromic-salts are usually green or violet in colour.

102. *AmHO:* pale bluish-green precipitate (Cr_2Ho_6): if AmHO is added in large quantity and the liquid is heated some of the precipitate is dissolved, producing a beautiful violet-red solution whose colour is best seen after filtering; but from this solution the Cr_2Ho_6 is reprecipitated on boiling the liquid for several minutes in a porcelain dish.

103. *KHO* added in small quantity gives the same precipitate (Cr_2Ho_6): if more cold KHO is added the precipitate is entirely dissolved to a green fluid, on diluting this liquid with water and boiling for several minutes the Cr_2Ho_6 is reprecipitated and the liquid becomes colourless: AmCl added to the KHO solution also reprecipitates the Cr_2Ho_6.

103 *a.* If to some of the green liquid, obtained by adding KHO in excess to the chromium solution, a little red-lead or lead-peroxide (PbO_2) is added and the liquid is then boiled, a yellow liquid is obtained from which a yellow precipitate ($PbCrO_4$) is thrown down by addition of $H\bar{A}$ in excess.

104. *Am_2S* precipitates Cr_2Ho_6, H_2S gas being given off or remaining dissolved. The reaction is similar to that with $AlAm(SO_4)_2$ par. 93.

105. *Blowpipe reaction.*—If Na_2CO_3 and KNO_3 be mixed with any solid substance containing Cr, and the mixture be fused on a piece of platinum foil or in a porcelain crucible or lid, or in a loop of platinum wire as was directed for the borax bead (31), a yellow mass is formed coloured by Na_2CrO_4. If this yellow mass is dissolved by boiling it with water, a yellow solution is obtained: this solution, if made acid with $H\bar{A}$ and boiled for a few minutes to drive off CO_2, gives with $Pb\bar{A}_2$ solution a yellow precipitate of $PbCrO_4$.

106. Cr forms two classes of compounds: in one class Cr in combination with oxygen functions as an acid radicle: this class includes the chromates such as K_2CrO_4 and $K_2Cr_2O_7$, they are usually yellow or reddish in colour; but Cr also forms a series of salts in which it acts as a metallic-radicle; these are usually green or violet, and give the above reactions for Cr. The latter compounds pass by oxidation *into the former*, as in reactions (103) and (105) where the oxidation is caused by PbO_2 and KNO_3 respectively. Chromates pass by reduction into the green compounds; examples of this change will be found under the tests for chromic acid (286). This reduction of chromic acid causes it to be detected in the course of analysis as a base, and a special experiment has to be performed to ascertain whether the Cr was present originally as a metallic or as an acid radicle.

107. GROUP III.A.—TABLE OF DIFFERENCES.

Tests.	Al—salts.	Fe'''—salts.	Fe''— salts.	Cr—salts.
a. For liquids.				
1. Add AmHO:	*White* flocculent precipitate.	*Reddish - brown* flocculent precipitate.	*Dingy-green* precipitate, rapidly turning brown when left exposed to the air.	*Pale-green* flocculent precipitate, colour unaltered by exposure to air.
2. Add KHO:	*White* flocculent precipitate, *easily soluble in excess of KHO.*	*Reddish - brown* flocculent precipitate, *insoluble in excess of KHO.*	The same precipitate as with AmHO, *insoluble in excess of KHO.*	*Pale-green* precipitate, *soluble in excess of cold KHO, but reprecipitated on diluting and boiling for some time.*
3. Add KCyS and a few drops of HCl:	——	*Deep blood - red* coloration, destroyed by pouring into $HgCl_2$ solution.	*No coloration* unless Fe''' is also present.	——
b. Blowpipe tests for solids.				
4. Fuse with Na_2CO_3 and KNO_3 on platinum foil:	——	On cooling, a white mass of Na_2CO_3 remains, with dark - brown particles of Fe_2O_3.	Same as Fe'''.	On cooling, a *yellow* mass remains, easily soluble in water; the solution, after having been boiled with excess of $H\bar{A}$, gives a yellow precipitate with $Pb\bar{A}_2$.
5. Heated on charcoal:	In the outer flame when cool if moistened with $Co(NO_3)_2$ solution and reheated strongly gives a *fine blue mass.*	In inner flame mixed with Na_2CO_3 gives a grey magnetic powder. (38a.)	Same as Fe'''.	——
6. Fused in borax bead:	——	*Reddish - yellow* in outer flame. *Greenish-yellow* in inner flame.	Same as Fe'''.	*Green* both in outer and inner flames.

Members of this group occurring singly are easily distinguished by one or other of the above tests, the first three serving for liquids, the last three for solids: tests 2 and 5 are the best for Al, 2, 4, and 6 for Cr, and 1, 3 or 6 for Fe.

108. (s) *The separation and detection of these three substances when mixed* depend upon the following differences:—

1. The solubility of Al_2Ho_6 in *boiling* KHO, in which Fe_2Ho_6 and Cr_2Ho_6 are insoluble.
2. The conversion of Cr_2Ho_6 by fusion with Na_2CO_3 and KNO_3 into soluble Na_2CrO_4: Fe_2Ho_6 remaining as Fe_2O_3 which is insoluble in water.
3. The yellow colour of the solution of Na_2CrO_4 in water, and production of a yellow precipitate by acidifying it with $H\bar{A}$ and adding $Pb\bar{A}_2$.
4. The blood-red coloration obtained by adding KCyS to the Fe_2O_3 dissolved in HCl.

109. (s) A solution which may contain Al, Fe, Cr, is examined in the following manner:—

To a few drops of the solution acidified with HCl add a little K_4FeCy_6; if a blue precipitate is produced Fe is present; this portion is rejected. Two other small portions of the acidified solution are then tested; one with $K_6(FeCy_6)_2$, which by giving a dark blue precipitate shows the presence of Fe''; the other with KCyS, which if it gives a blood-red colour proves the presence of Fe'''.

Boil the rest of the solution for several minutes, adding a few drops of strong HNO_3 if Fe'' is present; add some AmCl, then AmHO until the liquid after being well stirred is alkaline and smells of NH_3; boil, filter, and examine the precipitate by Table III.A (436), disregarding all the appended notes.

GROUP III.B.—ZINC GROUP.

110. This group includes Zn, Mn, Ni, Co; its members differ from those of Groups IV. and V., by being precipitated by Am_2S in neutral or alkaline solution, and by H_2S in *alkaline solution*: they are not precipitated by the group reagents for Groups III.A, II., and I. Mn is however liable to be precipitated by AmHO, even in the presence of AmCl, if the solution stands exposed to air for some time.

The members of this group show no characteristic flame colorations, but with the exception of Zn give characteristic colours to a borax bead.

$BaCO_3$ does not precipitate this group as it does Group III.A, unless its members are present as sulphates.

ZINC (Zn).—Use $ZnSO_4.7H_2O$.

Zinc salts are usually colourless.

111. *Am_2S:* white precipitate (ZnS): the precipitate often appears yellow from the presence of excess of yellow Am_2S in the liquid: its true colour is seen in the next reaction, or by letting the precipitate produced by Am_2S settle, or by filtering it. For the solubility of this precipitate see the end of the next reaction.

112. *H_2S*:* white precipitate (ZnS): the Zn is only partly precipitated from a neutral solution, since H_2SO_4 is separated and dissolves the ZnS :—

$$ZnSO_4 + H_2S = ZnS + H_2SO_4 ;$$

but if NaHO or AmHO be added to neutralise the H_2SO_4 when it is set free, the Zn may be entirely precipitated: addition of $Na\bar{A}$ also causes complete precipitation, since $H\bar{A}$ replaces H_2SO_4 in the solution, and ZnS is insoluble in $H\bar{A}$:—

$$ZnSO_4 + H_2S + 2Na\bar{A} = ZnS + Na_2SO_4 + 2H\bar{A}.$$

Add H_2S to some $ZnSO_4$ solution to which AmCl has been first added and then AmHO until it is alkaline, and show by adding to separate portions of this liquid HCl, $H\bar{A}$ and Am_2S, that ZnS is soluble in HCl, and insoluble in $H\bar{A}$ and in Am_2S†.

113. *KHO* added in small quantity gives a white precipitate ($ZnHo_2$): if more KHO is added, this precipitate dissolves;

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* H_2S may be used in this group as sulphuretted hydrogen water.

† The tests which show the solubility of the sulphides of this group are best tried on the precipitate obtained by adding AmCl, AmHO, and H_2S, unless freshly prepared colourless Am_2S can be obtained, since from yellow Am_2S acids precipitate sulphur.

the $ZnHo_2$ is however reprecipitated as such by adding much water to this solution and boiling it, or as ZnS by passing H_2S gas into it.

114. Solid $ZnSO_4.7H_2O$ powdered with Na_2CO_3 in a mortar, then heated on charcoal in the inner blowpipe flame, gives an incrustation on the charcoal, which is *yellow* whilst hot and white when cold. It cannot be driven away by the outer blowpipe flame, but is easily removed by the inner flame. If this incrustation on cooling is moistened with cobalt-nitrate solution and strongly heated in the outer blowpipe flame it becomes *green.*

The above changes of colour are more distinctly obtained by igniting a little solid $ZnSO_4$ in the outer blowpipe flame on charcoal, it is *yellow* whilst hot, and *white* when cold: if moistened with $Co(NO_3)_2$ solution and reheated in the outer flame, it becomes *green.*

MANGANESE (Mn).—Use $MnCl_2$ or $MnSO_4$.

Manganous salts are usually of a pale pink colour. Alkaline manganates are green, permanganates purple.

115. *Am_2S:* flesh-coloured or pale pink precipitate (MnS): the liquid should be filtered, since the precipitate often appears discoloured by the yellow Am_2S, the colour of the precipitate on the filter is then easily seen; the colour of the moist precipitate changes to dark brown on standing in the air upon the filter. For the solubility of this precipitate see (116).

116. *H_2S* precipitates MnS partly from neutral solutions, entirely from alkaline solutions, but not at all in presence of free HCl or $H\bar{A}$: show with separate portions of the MnS precipitate, obtained by adding AmCl, AmHO, and then H_2S solution to the liquid, that MnS is soluble in HCl and in $H\bar{A}$, but insoluble in Am_2S.

117. *KHO:* white precipitate ($MnHo_2$), quickly turned brown by pouring the liquid containing the precipitate into a white porcelain dish or upon a filter: the precipitate is insoluble in excess of KHO.

118. *AmHO* gives the same precipitate, but if sufficient AmCl is first added, AmHO produces no immediate precipitate, the solution however on standing exposed to the air turns brown and the Mn is gradually precipitated as brown Mn_2Ho_6.

118*a*. Pour a little $MnSO_4$ (not $MnCl_2$) solution upon some red or brown lead oxide (PbO_2), add HNO_3 which must be quite free from Cl, boil the mixture and allow the powder to settle: the clear liquid is coloured deep red by the formation of permanganic acid ($HMnO_4$). The production of this colour is prevented by the presence of even a minute quantity of chlorine.

119. *Blowpipe tests.*—If any solid substance containing Mn be fused in the outer flame with a mixture of Na_2CO_3 and a little KNO_3 upon platinum foil, a *bluish-green mass* is obtained on cooling: the mixture should be fused by heating the lower surface of the foil with the blowpipe flame. The test may be also made by fusing a mixture of Na_2CO_3 and KNO_3 into a bead in a loop of platinum wire, and then fusing the substance into this bead by heating it in the extreme tip of the outer flame.

119*a*. Fused with Na_2CO_3 on charcoal in the inner flame a grey magnetic powder is obtained (33 *a*).

120. *Borax bead.*—Use *very* little MnO_2* :—

In the outer flame $\left\{\begin{array}{l}\textit{Violet-red}\text{ bead whilst hot.}\\ \textit{Amethyst-red}\text{ on cooling.}\end{array}\right.$

In the inner flame.—A colourless bead.

NICKEL (Ni).—Use $NiSO_4.7H_2O$.

Nickel salts are usually bright green in colour.

121. *Am_2S:* black precipitate (NiS); add more *yellow* Am_2S† boil and filter, a brown filtrate runs through, coloured by NiS dissolved in the excess of Am_2S: pour this filtrate into a porcelain dish and boil for some time, adding distilled water if necessary to prevent the dish becoming dry; the black NiS will be precipitated and may be filtered off, giving a colourless filtrate. If $H\bar{A}$ is added to the dark filtrate until it is acid, the NiS is also precipitated from it.

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* A borax bead is often opaque from the use of too much substance. If such is the case fuse the bead in the blowpipe flame, then by a sudden jerk throw some of the fused mass off, and fuse again the remaining portion with fresh borax: repeat this if necessary. The fused bead often becomes transparent when flattened by squeezing with small pincers.

† The Am_2S must be yellow; colourless Am_2S does not dissolve NiS.

122. *H_2S:* black precipitate (NiS) in neutral solutions or in solutions acid with $H\bar{A}$ (112), but HCl prevents the precipitation; show with portions of the liquid containing NiS, obtained by adding H_2S to $NiSO_4$ solution to which a little AmCl and AmHO have been added, that NiS is insoluble in cold dilute HCl and in $H\bar{A}$, but soluble when heated with HCl after adding a crystal of $KClO_3$.

123. *KHO:* light green precipitate ($NiHo_2$).

124. *AmHO* added in very small quantity: bluish-green precipitate ($NiHo_2$), soluble in excess of AmHO to a violet-blue liquid, soluble also in AmCl. If AmCl be first added AmHO causes no precipitate.

125. *KCy* solution* added in small quantity gives a yellowish-green precipitate ($NiCy_2$): by further addition of KCy this precipitate is dissolved, but HCl reprecipitates $NiCy_2$ from the solution.

126. If some $NiSO_4$ solution is made acid with several drops of HCl, then KCy solution gradually added, whilst stirring or shaking the liquid, until the precipitate is redissolved, and the solution thus obtained is boiled for a short time, then cooled and divided into two parts: HCl added in excess to one part will produce a precipitate of $NiCy_2$, often only appearing after a time: Na_2Cl_2O, or Br-water after excess of NaHO, added to the other part will give on warming a black precipitate (Ni_2Ho_6).

Blowpipe reactions:—

127. Solid $NiSO_4.7H_2O$ powdered with Na_2CO_3 in a mortar and then fused on charcoal in the inner blowpipe flame, leaves a grey powder which is attracted by the magnet. (33 *a.*)

128. *Borax bead.*

In outer flame	*Violet* or *sherry-red* bead whilst hot. *Pale yellow* on cooling.
In inner flame, after being heated for some time.	*Black* or *opaque* bead.

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* KCy solution must always be prepared immediately before use: it is decomposed by keeping.

Cobalt (Co).—Use $Co(NO_3)_2.6H_2O$.

Cobalt salts are usually reddish-pink in colour.

130. *Am_2S:* black precipitate (CoS); on adding much Am_2S, boiling and filtering, the filtrate is coloured yellow by Am_2S and is not dark coloured, since CoS is insoluble in Am_2S (difference from NiS). For the solubility of CoS see Exp. 131.

131. *H_2S:* black precipitate (CoS), only forms in alkaline solutions or in solutions acid with $H\bar{A}$ (112); HCl prevents the precipitation. Add AmCl then AmHO in excess, and then H_2S-water to some $Co(NO_3)_2$ solution, and pour into separate portions of this liquid HCl and $H\bar{A}$, the precipitate does not dissolve; to the portion containing HCl add a crystal of $KClO_3$ and heat, the precipitate dissolves readily.

132. *KHO:* blue precipitate ($CoHo_2$), turning green if poured out upon a watch glass and allowed to stand in the air, and becoming pale red on being boiled; the red colour is, however, frequently more or less masked by a brown cobalt hydrate precipitated at the same time.

133. *AmHO:* bluish-green precipitate, having the same properties as that given by KHO: soluble in AmCl, hence if AmCl is added before AmHO no precipitate is produced.

134. *KCy* solution added in small quantity gives a reddish-brown precipitate ($CoCy_2$): add more KCy solution slowly and whilst shaking the liquid, the precipitate dissolves; now add HCl, the $CoCy_2$ is reprecipitated.

135. If some $Co(NO_3)_2$ solution is made acid with a few drops of $H\bar{A}$, then KCy solution added slowly until the precipitate at first formed just redissolves, and the liquid is boiled until no further smell of HCy is evolved, then cooled and divided into three parts, it will be found that neither HCl, nor Na_2Cl_2O, nor Br-water with excess of NaHO solution, produces a precipitate on warming the liquid. [Difference from Ni (126).]

The difference of behaviour of the Ni and Co solutions which have been boiled with excess of KCy, is due to the fact that $NiCy_2$ forms with KCy a feeble compound ($NiCy_2.2KCy$) which is soluble in water, but is easily decomposed: whereas $CoCy_2$ forms with KCy in the air the very stable soluble compound $K_6(CoCy_6)_2$, "Potassium cobalticyanide."

Blowpipe reactions:—

136. Fused with Na_2CO_3 on charcoal in the inner flame, $Co(NO_3)_2$ gives a grey metallic powder attracted by the magnet (33 *a*).

137. *Borax bead:* strong solution may be employed, the bead being dipped into it: *fine blue* bead in both inner and outer flames: opaque if too much $Co(NO_3)_2$ has been used.

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138. When a solution is to be examined for only one member of this group, its detection by one or more tests in the table (139) will be very easy. The precipitate with

39. GROUP II

Tests.	Zn—salts.	Mn—salts.
a. For liquids.		
1. Add Am_2S, or better, AmCl, AmHO in excess, and H_2S-water.	*White precipitate:* soluble in cold dilute HCl: *insoluble in* $H\overline{A}$.	*Pink precipitate:* solubl cold dilute HCl: *solubl* $H\overline{A}$.
2. Add KHO:	*White precipitate: soluble in excess of KHO.*	*White precipitate;* turn brown in the air: *insolubl* KHO.
3. Add KCy solution: This test need only be tried when a *black* precipitate has been obtained by Test No. 1.	——	——
b. Blowpipe tests for solids.		
4. Fused with Na_2CO_3:	On charcoal in the inner flame: gives a white incrustation, which, if moistened with $Co(NO_3)_2$ and heated in the outer flame, turns *green.* The substance itself, when strongly heated after having been moistened with $Co(NO_3)_2$ also becomes green.	On charcoal in the in flame: a grey magn powder (33 *a*). On platinum foil in ou flame: a *bluish-green* m showing more rapidly i little KNO_3 be mixed w the Na_2CO_3 before fusing.
5. Borax bead:	——	Outer flame.—*Amethyst-re* Inner flame.—*Colourless.*

Am_2S is distinctive, ZnS and MnS being easily recognised by their different colours; NiS and CoS, which differ from ZnS and MnS by being black, are distinguished from one another by the solubility of NiS in excess of Am_2S, the liquid therefore giving a brown filtrate. KHO also gives characteristic precipitates with each of these substances. The most distinctive tests for Ni and Co are 3 and 5; for Zn and Mn, 2, 4, and 5.

140. (*s*). *The method for separating and detecting Zn, Mn, Ni, Co, when mixed,* depends on :—

1. The solubility of NiS in yellow Am_2S; ZnS, MnS and CoS being insoluble*. NiS is precipitated from this solu-

LE OF DIFFERENCES.

Ni—salts.	Co—salts.
ck precipitate: soluble in boiling yellow *m_2S** to a dark solution: insoluble in cold lute HCl and in $H\bar{A}$.	*Black precipitate: insoluble in boiling yellow Am_2S**, in cold dilute HCl, and in $H\bar{A}$.
en precipitate: insoluble in KHO.	*Blue precipitate*: insoluble in KHO.
led in excess to the slightly acid solution d boiled for some time, then Na_2Cl_2O (or r-water and excess of NaHO) added, gives black precipitate on heating.	Added in excess to slightly acid solution and boiled for some time, Na_2Cl_2O (or Br-water and excess of NaHO) gives no precipitate on heating.
charcoal in the inner flame, a grey magnetic powder (33 *a*).	Same as Ni.
ter flame. { *Violet* or *yellow* while hot. *Yellow* when cold. } ner flame.—*Grey* or *opaque* bead.	Outer and inner flames.—*Fine-blue* bead.

In order to ascertain whether the precipitate has been dissolved, filter and rve whether the filtrate is dark in colour.

tion by boiling or by addition of $H\bar{A}$, and the presence of Ni is then confirmed by fusion in a borax bead.

2. The solubility of ZnS and MnS in cold dilute HCl, NiS and CoS being almost insoluble. (Note 1, below).

3. The solubility of $ZnHo_2$ in cold KHO, in which $MnHo_2$ is insoluble; white ZnS is then precipitated from this solution by H_2S.

4. The production of a bluish-green mass by fusing $MnHo_2$ with Na_2CO_3 and KNO_3.

5. The difference of behaviour of the KCy solution of Ni and Co with Na_2Cl_2O, or with Br-water and excess of NaHO: and the marked difference in their borax beads. (Note 2, below).

Note 1.—Mn may also be separated from Ni and Co by passing H_2S into the solution containing $Na\bar{A}$ (112): Mn remains in solution, NiS and CoS are precipitated.

Note 2.—A modification by Henry of Rose's method for separating Ni and Co is also to be recommended; it depends upon the fact that, whilst Co is precipitated as Co_2Ho_6 by $BaCO_3$ in presence of Br, Ni remains in solution. The sulphides are dried and then strongly ignited in an open porcelain crucible: the metals remaining as oxides are dissolved by boiling with a little strong HCl, carbon is filtered off if necessary, and to the clear solution, after being cooled and mixed with Br-water, excess of $BaCO_3$ is added in a small corked flask; the flask is then well shaken and allowed to stand for about half an hour: on filtering, Co if present is detected in the precipitate by the borax bead: Ni is precipitated from the filtrate by heating it with KHO; on filtering, the presence of Ni in the precipitate may be confirmed by fusing a portion of it in the borax bead.

141. (S) A solution is examined for Zn, Mn, Co, Ni, by adding AmCl to it in a boiling tube, then AmHO if necessary until it is alkaline, and then Am_2S until, after being well stirred or shaken, the liquid smells of it. The liquid is then boiled, a few drops are poured upon a filter and the colour of the filtrate noted; if it is colourless, sufficient Am_2S has not been added, more Am_2S is then to be poured in, and the liquid again boiled. When a few drops run through the filter brown or yellow in colour, Am_2S has been added in excess, the whole is boiled and filtered and the precipitate examined by Table III.B (437): the filtrate if yellow is rejected, if brown it is examined for Ni according to (431).

GROUP II.A.—COPPER GROUP.

142. This group includes Hg'', Pb, Bi, Cu, Cd; its members differ from those of Groups III.A, III.B, IV. and V., in being precipitated by H_2S in acid solutions. With the exception of Pb, which in a strong solution is partially precipitated by HCl, the members of this group are not precipitated by HCl.

The sulphides differ from those of Group II.B, by being insoluble in Am_2S or KHO. If very much acid is present, H_2S does not precipitate the members of this group readily or completely, they are precipitated however on dilution.

MERCURICUM (Hg'').—Use $HgCl_2$.

143. Hg forms two series of compounds distinguished as *mercuric* and *mercurous*: the mercury in these two states behaves differently with reagents, and may be distinctively called *mercuricum* and *mercurosum*: in the latter state it is classed under Group I.

144. *H_2S**: black precipitate (HgS): if the H_2S solution is added slowly, the precipitate is first *white*, then *brown* or *orange*, and ultimately becomes *black*: these changes of colour during the addition of H_2S are characteristic of mercuric salts. Filter and wash the precipitate: place portions if it in three test-tubes: boil one portion with strong HNO_3 and another with strong HCl, the precipitate is not dissolved by either acid: mix the contents of the two tubes and warm again, the precipitate will now dissolve: boil the third portion with Am_2S, the precipitate does not dissolve. Hence HgS is insoluble in hot strong HNO_3, in hot strong HCl, and in Am_2S, but is easily soluble in a mixture of HNO_3 and HCl or "aqua regia".

145. *$SnCl_2$:* white precipitate (Hg_2Cl_2): if more $SnCl_2$ is added and the liquid boiled, the white precipitate becomes grey and consists of fine particles of Hg; if the liquid be decanted and the grey precipitate be then boiled with strong HCl, the Hg particles unite to globules visible by a lens or frequently by the naked eye.

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* A test-tube half full of the solution must be used in order to get sufficient precipitate for the experiments below. This remark applies also to the liquid used for obtaining the H_2S-precipitate with the other members of this group.

$$2HgCl_2 + SnCl_2 = Hg_2Cl_2 + SnCl_4.$$
$$Hg_2Cl_2 + SnCl_2 = 2Hg + SnCl_4.$$

146. *Cu:* if a small strip of copper sheet or a copper coin, whose surface has been polished and cleansed by rubbing it with sand paper, be immersed in some $HgCl_2$ solution made acid with a drop or two of HCl, it will soon become coated with a grey film of Hg:—

$$HgCl_2 + Cu = Hg + CuCl_2.$$

If the surface of the Cu, after having been immersed for several minutes, is dried and rubbed hard with a cloth or piece of wash-leather, it will appear more or less whitened like silver, the liquid metal Hg having "amalgamated" the Cu. The Hg may be readily driven off by heating the Cu strongly; if this heating is performed in a test-tube, or better in a small hard glass tube sealed at one end (10), a sublimate of globules of Hg forms upon the cool sides of the tube.

147. Mix *a little* solid $HgCl_2$ intimately with about three times as much Na_2CO_3, by powdering them together in a mortar, or with a pestle on a watch glass. Pour some of this dried mixture into a perfectly dry small test-tube (see note below), and cover the mixture with a small layer of Na_2CO_3; now heat strongly first the layer of Na_2CO_3, then gradually the mixture: Hg will sublime, condensing in globules on the sides of the tubes. Sometimes the globules are so small that the layer appears as a grey film; they may be united into visible globules by rubbing the film with a splinter of wood or with a thin glass rod (11 *a*), or the separate globules may be seen by means of a pocket lens.

Note.—The powdered mixture is best dried by spreading it out upon a watch glass and placing it in a steam or water oven; it may be dried also by supporting it on the filter dryer (25) at some height above a *small* flame. A powder such as the above is most easily placed in a narrow closed tube by alternately scooping the powder up with the mouth of the tube, and tapping the tube so as to shake the powder to the bottom: or by turning the powder out upon a piece of note paper folded over into a trough and pouring it thence into the tube, holding both over the watch glass to catch any substance which falls. Both the inside of the tube and the powder must be perfectly dry, else some powder obstinately adheres to the sides of the tube and obscures the sublimate. The heating must never be commenced until the sides of the tube are perfectly clean: they are cleansed if necessary with a twisted slip of filter-paper or a wooden match; also if any drops of water condense on the inside of the tube during the first stage of the heating they must be removed by a twisted piece of filter-paper, or by a small piece of filter-paper rolled round a wooden match or a thin piece of wire.

148. Heat a little solid $HgCl_2$ in a tube closed at one end; the substance sublimes in white fumes, since Hg-compounds are volatile.

LEAD (Pb).—Use $Pb\bar{A}_2.3H_2O$ dissolved in water to which a little $H\bar{A}$ has been added.

149. *H_2S:* black precipitate (PbS): this precipitate is red if much hydrochloric acid is present in the solution, but becomes black on diluting with water and passing H_2S, or on adding more H_2S-solution. Filter or decant, and show with separate portions of the precipitate that PbS is insoluble in KHO or Am_2S, soluble in boiling dilute HNO_3, but converted by boiling strong HNO_3 into insoluble $PbSO_4$.

150. *HCl:* white precipitate ($PbCl_2$), forms only in cold and strong solutions: on boiling, the precipitate dissolves*, but the $PbCl_2$ separates again in beautiful crystals on cooling.

151. *H_2SO_4:* white precipitate ($PbSO_4$): this precipitate is much less soluble in dilute H_2SO_4 than in water, hence H_2SO_4 should be added in excess to a pretty strong solution of Pb: pour off into two test-tubes and let the liquid stand; decant the liquid from the precipitate, and show that the precipitate may be dissolved by pouring upon it $H\bar{A}$ or

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* If the precipitate does not entirely dissolve, add a little water and boil again.

$H_2\bar{T}$, then AmHO in excess, and boiling; show also that it dissolves in boiling strong HCl.

152. *K_2CrO_4 or $K_2Cr_2O_7$:* yellow precipitate ($PbCrO_4$); pour off into two tubes and show that the precipitate is soluble in KHO, but insoluble in $H\bar{A}$.

153. *Blowpipe reaction.*—Mix well some powdered $Pb\bar{A}_2.3H_2O$ with about twice as much Na_2CO_3; heat the mixture in a cavity on a piece of charcoal in the inner flame; a yellow incrustation will form around the cavity and small bright white globules of Pb will be seen within it. The incrustation is readily removed when heated in the inner blowpipe flame, colouring the flame azure-blue. Detach one of the globules with the point of a knife, place it on the bottom of a mortar turned upside down and give it a smart blow with the pestle; it flattens out without breaking into powder, because Pb is *malleable* not *brittle.* If one of the globules be fixed upon the point of a penknife, it will be found by gentle friction on paper to mark it as a blacklead pencil does.

BISMUTH (Bi).—Use $Bi(NO_3)_3.5H_2O$ dissolved in dilute HCl.

154. *H_2S:* brownish-black precipitate (Bi_2S_3): let stand, and decant most of the water; then pour some of the precipitate into three test-tubes, and show that it is insoluble in Am_2S and in KHO, but soluble in strong HNO_3.

$$2Bi(NO_3)_3 + 3H_2S = Bi_2S_3 + 6HNO_3.$$

155. *AmHO or KHO:* white precipitate ($BiHo_3$); pour off a small quantity of the liquid and precipitate into a tube, add much AmHO and warm, the precipitate is undissolved; filter off the rest of the precipitate and dissolve it by pouring upon the filter *a few drops* of hot dilute HCl: keep this solution.

156. *H_2O:* pour the HCl solution obtained from the last reaction, or some of the original solution, into a large quantity of distilled water contained in a beaker: on stirring and letting stand a milkiness appears, due to the formation of BiOCl:

$$BiCl_3 + H_2O = BiOCl + 2HCl.$$

Pour a little of the milky liquid into two test-tubes; warm one portion after adding to it a little strong HCl, the precipitate dissolves: to the other add $H_2\overline{T}$ and warm, the milkiness remains (difference from SbOCl). If but little Bi is present this milkiness often only appears after stirring and letting the liquid stand for five or ten minutes.

157. *K_2CrO_4 or $K_2Cr_2O_7$* (see Note below): yellow flocculent precipitate [$Bi_2(CrO_4)_3$]: add KHO, the precipitate does not dissolve (difference from $PbCrO_4$); it dissolves completely in HCl or HNO_3 added in excess.

Note.—Since this precipitate is soluble in HCl, and HCl is present in the Bi-solution used, the free HCl must first be removed by dissolving in a little of the Bi-solution a sufficient quantity of solid $Na\overline{A}$; the free HCl is thus replaced by $H\overline{A}$, which does not dissolve the precipitate:—

$$Na\overline{A} + HCl = NaCl + H\overline{A}.$$

158. *H_2SO_4:* no precipitate.

159. *Blowpipe reaction.*—A mixture of powdered $Bi(NO_3)_3$ and Na_2CO_3 heated on charcoal in the inner flame, gives an incrustation *orange-red* whilst hot, *yellow* when cold; also white globules of Bi which are very brittle, being crushed to powder by a sharp blow with a pestle (153).

COPPER (Cu).—Use $CuSO_4.6H_2O$.

Copper salts are usually blue, sometimes green in colour; their colour commonly becomes very pale or disappears when the salts are thoroughly freed from water of crystallisation.

160. *H_2S:* brownish-black precipitate (CuS): filter, keeping the funnel carefully covered with a glass plate, as air quickly oxidises CuS to $CuSO_4$ which dissolves and runs through with the filtrate. Place some of the precipitate in five test-tubes and show that it is insoluble in KHO, in Am_2S, and in boiling dilute H_2SO_4, but dissolves in boiling HNO_3 dilute or strong, and in KCy solution.

161. *AmHO* added in very small quantity: a *greenish-*

blue precipitate; if more AmHO is added this precipitate dissolves, yielding an *intensely blue* liquid containing $(N_2CuAm_4H_2)SO_4$ which becomes again light-blue on adding an acid in excess.

162. *H_2SO_4:* no precipitate.

163. *K_4FeCy_6:* reddish-brown precipitate (Cu''_2FeCy_6), insoluble in $\overline{HA}$; the colour is best seen by trying the reaction in a white porcelain dish. In *very* dilute solution, only a reddish colour is produced.

164. *Fe:* a bright strip of steel or iron, such as the blade of a penknife, freed from grease by rubbing with sand-paper or by boiling in a little dilute KHO and washing, when dipped into $CuSO_4$ solution made acid with a few drops of H_2SO_4, is covered with a red film of Cu after a time.

165. *Zn and Pt:* Zn if placed upon a piece of platinum foil or wire in some $CuSO_4$ solution contained in a porcelain dish or watch glass, causes a red film of Cu to deposit on the platinum: the $CuSO_4$ solution should be first made acid with a few drops of H_2SO_4.

Blowpipe reactions:—

166. A mixture of powdered $CuSO_4.6H_2O$ and Na_2CO_3 if heated on charcoal in the inner blowpipe flame, gives red scales of Cu, which are best seen by separating them as directed in (33*a*.)

167. *Borax bead.*	In outer flame:	*Green* whilst hot, *blue* when cold.
	In inner flame:	*Red* or *colourless* bead when cold, obtained only when very little Cu is present, and the bead is long heated in a good reducing flame*.

168. *Flame coloration:* a loop of platinum wire dipped into $CuSO_4$ solution, and held in the inner blowpipe flame, or in the Bunsen flame gives a *green* coloration; the flame shows a *blue centre*, after the loop has been moistened HCl.

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* The red bead is much more easily and rapidly obtained by fusing a fragment of Sn or Zn into the bead, the former gives a clear, the latter a *turbid bead*.

CADMIUM (Cd).—Use $CdSO_4.4H_2O$.

169. *H_2S:* bright yellow precipitate (CdS): boil off the H_2S, and pour the liquid into several tubes; show with these separate portions that the precipitate is insoluble in Am_2S, in KHO, and in KCy solution, but soluble in boiling dilute HNO_3 and in boiling dilute H_2SO_4: dilute the H_2SO_4 solution with much water and pass H_2S for a short time, yellow CdS is reprecipitated.

170. *AmHO* added in small quantity, best by using AmHO much diluted: white precipitate ($CdHo_2$); on adding more AmHO the precipitate readily dissolves.

171 *H_2SO_4*: no precipitate.

172. *Blowpipe reaction.*—Powdered $CdSO_4$ mixed with Na_2CO_3 and heated on charcoal in the inner blowpipe flame, gives a brown incrustation.

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173. GROUP II.A.—TABLE OF DIFFERENCES.—(See pp. 108-109.

174. The colour of the H_2S precipitate, and its behaviour with strong HNO_3, enable the analyst to detect one member of this group when present alone: the most characteristic special tests for each member of this group will be found under its reactions.

175. (s) The separation of Hg, Pb, Bi, Cu, Cd, depends upon the following differences:—

1. The solubility of Bi_2S_3, CuS, CdS in strong boiling HNO_3; HgS and PbS being insoluble, and remaining as HgS and $PbSO_4$.

2. The solubility of $PbSO_4$ in $H\bar{A}$ and excess of AmHO; HgS being insoluble.

3. The solubility of $CuHo_2$ and $CdHo_2$, and insolubility of $BiHo_3$, in excess of AmHO.

4. The solubility of CdS and insolubility of CuS in boiling dilute H_2SO_4. The insolubility of CdS in KCy solution, in which CuS easily dissolves, furnishes another means of separation.

After having been separated, the presence of each metal is confirmed by some special test.

176. (s) *A solution which may contain Pb, Hg, Bi, Cu, Cd, mixed* is first made acid, if not already so, with a little HCl*; H_2S is then passed in a rapid stream through the solution for about five minutes; the precipitate is filtered off, the filtrate mixed with H_2S-water, or diluted with a little water and H_2S again passed for a short time, to ascertain whether

GROUP II.A.

Tests.	Hg—salts.	Pb—salts.
a. For liquids.		
1. Pass H_2S, or add H_2S water:	*Black* precipitate; when filtered and well washed, insoluble in boiling strong HNO_3, and *unchanged* by it.	*Black* precipitate; almost insoluble in boiling strong HNO_3, changed by it into *white* $PbSO_4$.
2. Add H_2SO_4:	——	White precipitate.
3. Add AmHO:	*White* precipitate, insoluble in excess.	*White* precipitate, insoluble in excess.
b. Blowpipe reactions for solids.		
4. Heated with Na_2CO_3 on charcoal in the inner blowpipe flame:	Test for Hg by (147).	*Yellow* incrustation; white *malleable* globules.

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* A white precipitate of $PbCl_2$, which may form if the solution is very strong, is neglected, since it is readily convertible into PbS by H_2S.

all the metals have been completely precipitated: if no further precipitate is produced the filtrate may be rejected; but in case H_2S causes further precipitation, the gas must be passed for some time longer, and the liquid poured again through the filter, the filtrate only being rejected when H_2S no longer produces any precipitate in it; the precipitate is then examined by Table II. (435), commencing at Group II.A. (Copper Group), and using only the left-hand side of the Table.

TABLE OF DIFFERENCES.

Bi—salts.	Cu—salts.	Cd—salts.
Black precipitate; soluble in boiling strong HNO_3 to form a colourless solution.	*Black* precipitate; soluble in boiling concentrated HNO_3 to form a blue solution; insoluble in boiling dilute H_2SO_4.	*Yellow* precipitate; soluble in boiling HNO_3, and in boiling dilute H_2SO_4.
White precipitate, insoluble in excess. Confirm by (156).	*Blue* precipitate, easily soluble in excess to a deep blue liquid.	*White* precipitate, easily soluble in excess.
Orange-red incrustation; white *brittle* globules.	No incrustation; red metallic scales. Confirm by (167, 168).	*Brown* incrustation; no globules.

GROUP II.B.—ARSENIC GROUP.

177. This group includes As, Sb, Sn; its members differ from those of Groups III.A., III.B., IV. and V. by being precipitated by H_2S in a solution made acid with HCl; they differ from those of Group I., in not being precipitated by HCl.

The sulphides of this group, which are precipitated by H_2S, differ from those of Group II.A. by being soluble in Am_2S or KHO.

178. Each member of this group forms two series of compounds which resemble each other in many reactions, but also present several differences. The two classes are distinguished conveniently by the terminations *-ic* and *-ous* thus we speak of arsen*ic* and arseni*ous* acid.

ARSENIC (As).
- For arseni*ous* compound use HCl solution of As_2O_3.
- For arsen*ic* compound use water solution of $Na_2HAsO_4.12H_2O$.

A. TESTS WHICH GIVE ULTIMATELY THE SAME RESULT WITH BOTH SETS OF COMPOUNDS.

The two solutions should be taken in separate test-tubes and a portion of each tried in succession with each reagent; the difference of behaviour is thus more easily appreciated and remembered.

Arsen*ic* compounds are converted into arseni*ous* by boiling with HCl; this explains reactions (179) and (180) with arsen*ic* solutions.

Reagent.	Arsen*ious* solution.	Arsen*ic* solution.
179. H_2S :	In neutral solutions only a yellow colour is produced, but if the solution is acid with HCl a light yellow flocculent precipitate (As_2S_3) forms immediately even in the cold. Shake up and pour some of the liquid into another test-tube and show that KHO and Am_2S, added to the separate portions drop by drop, dissolve the precipitate, which, however, is thrown down again on addition of HCl in excess*.	No precipitate is produced in the cold solution acidified with HCl ; but if the acid solution is boiled, H_2S causes in the hot solution, first a milky precipitate of S, then yellow As_2S_3.
180. Cu : three or four narrow strips brightly polished with sand paper.	*Reinsch's test.*—If boiled in the solution to which some dilute HCl has been added, Cu becomes coated with a black film (As_2Cu_5) ; if the Cu strips are carefully dried by being pressed between filter-paper, and are heated in a small dry test-tube, a white sublimate of As_2O_3 forms in the upper part of the tube ; on being examined with a lens this is seen to consist of white octahedral crystals. This sublimate may be dissolved when cold in a little hot water, and the presence of As confirmed by tests (179) and (187). The presence of As in the deposit should always be confirmed in this way, since other metals besides As are deposited on Cu as a black film.	The same deposit is obtained as with As_2O_3, but only when strong HCl is added and the liquid boiled.

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* The KHO solution of the precipitate and the As_2S_3 precipitated from it by HCl are often discoloured by black sulphides formed by the action of H_2S upon Pb or Fe present as an impurity in the KHO : this discoloration may be almost entirely prevented by boiling the liquid before adding KHO, the free H_2S is thus expelled.

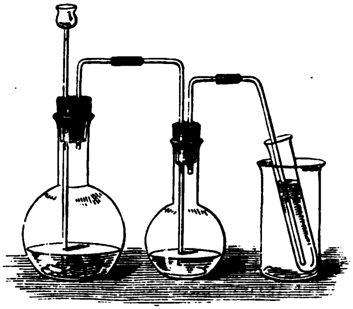
TESTS DEPENDING ON THE FORMATION OF AsH_3.

Note 1.—As AsH_3 is a very poisonous gas, the following experiments should be performed in a cupboard provided with a good draught, or in the open air.

Note 2.—Arsen*ic*-compounds give these reactions less readily than arseni*ous*, but by being boiled with HCl for a short time they are converted into arseni*ous* compounds, which then readily give the following reactions.

181. *Hofmann's method.*—If some scraps of Zn and some dilute HCl, both free from As, be placed together in a small flask fitted with a funnel-tube dipping below the liquid and a delivery-tube, as shown in figure 33*, H will be given off either at once or on adding a little strong HCl:—

FIG. 33.



$$Zn + 2HCl = 2H + ZnCl_2.$$

The hydrogen can be made to bubble through some $Pb\bar{A}_2$-solution contained in a second small flask connected with the first by a piece of india-rubber tubing; the gas, thus freed from any H_2S with which it might possibly have been mixed, is then allowed to bubble through some $AgNO_3$ solution contained in a test-tube which is supported in a small beaker; if the Zn and acid were free from As, no precipitate or colour is produced in the $AgNO_3$ solution.

If *a little* As_2O_3 solution be now poured into the larger flask through the funnel, a black precipitate (Ag) will begin to form in the $AgNO_3$ solution; this is produced by the AsH_3 evolved by the action of the H upon the As_2O_3:—

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* In order to be sure that the corks and tubes fit air-tight, moisten the outside of the cork after it has been placed in the neck of the flask, and having closed one tube, blow down the other. No air bubbles must be seen or heard to escape. Several of these apparatus may be kept ready fitted in the laboratory for general use.

$$As_2O_3 + 12H = 2AsH_3 + 3H_2O.$$

The AsH_3 on passing into the $AgNO_3$ solution, reacts thus:—

$$AsH_3 + 6AgNO_3 + 3H_2O = 6Ag + H_3AsO_3 + 6HNO_3.$$

Hence Ag is precipitated, and H_3AsO_3 remains in solution.

As soon as a copious precipitate has been obtained, the test-tube is removed. It will be best to put by the test-tube with its contents to be examined afterwards as directed below, and whilst the gas is coming off to proceed at once to use it for reactions (182) and (183).

The liquid in the test-tube is filtered, the precipitate rejected, and several drops of $AgNO_3$ solution are added to the clear filtrate. On mixing with this liquid drop by drop some very dilute AmHO,* and stirring or shaking after each addition, a light yellow precipitate (Ag_3AsO_3 or Ag_2HAsO_3) will be obtained. The formation of this precipitate on addition of AmHO, is explained by the fact that H_3AsO_3 is in the solution in the presence of $AgNO_3$ and also of HNO_3 (see last equation); now $AgNO_3$ forms with H_3AsO_3 a yellow precipitate (Ag_3AsO_3) if no free acid is present in the liquid (187); hence on neutralising with AmHO the free HNO_3 already present, and that which is formed by the action of $AgNO_3$ on H_3AsO_3, the precipitate appears:—

$$H_3AsO_3 + 3AgNO_3 + 3AmHO = Ag_3AsO_3 + 3AmNO_3 + 3H_2O.$$

This precipitate is also readily soluble in AmHO; hence great caution is requisite to use very dilute AmHO, and to add it gradually.

Caution.—Both flasks should be rinsed out each time after use, else some of the AsH_3 may remain in them and be driven out in a future operation.

182. Disconnect the larger flask from the smaller, and slip into the india-rubber joint a tube of hard glass about four inches in length whose end is drawn out to a fine jet, and supported on the ring of the retort-stand (fig. 34, p. 114); if the gas is not coming off briskly pour in through the funnel-tube a little strong HCl and light the gas as it issues from the jet[*]. The flame will differ from that of pure H by burning with a bluish tinge, and by giving off white fumes of As_2O_3[†].

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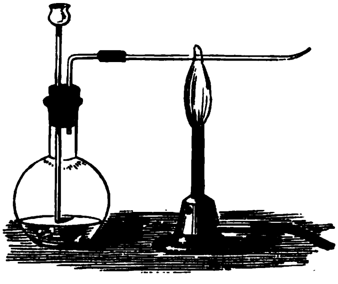
* Made by pouring several drops of AmHO solution into a test-tube, then nearly filling up with distilled water and shaking the tube.

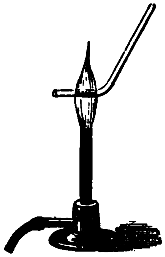
FIG. 34.



Press down upon the flame the inside of a porcelain crucible lid or of a porcelain crucible or dish, a dusky black film of As will be deposited upon the cool surface; pour upon this some Na_2Cl_2O solution, the stain is rapidly dissolved. Warm also a stain of As, obtained inside a porcelain dish, with Am_2S, it will dissolve and on gentle evaporation will leave *yellow* As_2S_3.

183. Add a little more strong HCl if necessary to cause the gas to come off rapidly, light it at the jet and heat a point near the middle of the tube strongly (see fig. 34); a black mirror of As will form just beyond the part heated by the flame, and the flame burning at the jet will meanwhile become colourless, showing that the arsenic has been removed from the hydrogen. Heat another point in the tube so as to produce a second mirror; then divide the tube between the mirrors, and show by warming one portion when cold with some Na_2Cl_2O solution in a test-tube, that the mirror is soluble in Na_2Cl_2O; show that the other, when heated in the tube held obliquely

FIG. 35.



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[*] If the experiment is performed as here directed, the H will have been evolved for some time before being lighted, and there is no fear of an explosion occurring; but if the H is being produced for the first time, allow it to escape briskly for four or five minutes, and ascertain that a test-tube full of the gas burns quietly (see Exp. 8, p. 10) before adding the liquid to be tested for As and lighting the gas at the jet.

[†] If these appearances are not noticeable, pour a little more As_2O_3 solution into the flask through the funnel and mix by gentle shaking.

in the flame, or bent as shown in fig. 35, gives a sublimate of As_2O_3. Dissolve the sublimate when cool in a little hot water and confirm the presence of As by reactions (179) and (187).

Tests (182) and (183) were first suggested by ***Marsh*** ; the former is usually known as ***Marsh's test.***

183 *a*. AsH_3 produces a characteristic colour with $HgCl_2$ solution. Remove the cork from the larger flask and push a plug of cotton-wool into its neck to stop any spirting from the liquid in the flask, then cover the mouth of the flask with a small piece of filter-paper moistened with $HgCl_2$ solution. The paper becomes coloured first *yellow* then *brown.*

184. If some pieces of Zn or Al be boiled in KHO solutions H is evolved which is free from odour, but if a little As_2O_3 solution be now added and the liquid be again boiled, a garlic odour is evolved owing to the formation of AsH_3 :—

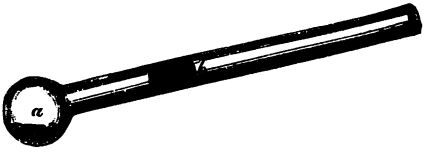
$$\left\{\begin{array}{l} Zn+2KHO=H_2+ZnK_2O_2 . \\ As_2O_3+12H=2AsH_3+3H_2O . \end{array}\right.$$

If a small slip of filter-paper moistened with $AgNO_3$ solution and placed on the end of a glass rod*, be held in the gas in the mouth of the tube whilst the liquid is being boiled, the paper will be stained black by the separation of Ag (181).

This reaction does not succeed with arsen*ic* compounds unless they are first reduced by boiling with HCl. It is not given by any Sb compounds (difference between As and Sb).

185. If any solid substance containing As be mixed with powdered Na_2CO_3 and KCy or with $K_2C_2O_4$ in a mortar, and a small quantity of the mixture, after having been dried for some time at a gentle heat on a watch glass, be introduced into a little bulb-tube of hard glass (fig. 36) or small test-tube at least three inches in length ; and if the substance be then heated, observing the precautious stated in the note under (147),

FIG. 36.



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* A simple method of getting a moistened piece of paper on the rod is to dip the rod into $AgNO_3$ solution ; then gently press the paper slip upon it whilst the rod is wet.

a black mirror of As forms in the cooler part of the tube and frequently a smell of garlic may be detected at the mouth of the tube. If the bulb be cut off and the mirror be heated by holding the tube obliquely in the flame (fig. 35), or if the As be resublimed in the test-tube, the mirror is converted into a sublimate of As_2O_3 crystals.

186. *Blowpipe reaction.*—If any solid substance containing As is mixed with Na_2CO_3 and the mixture is heated on charcoal in the inner blowpipe flame, a smell of garlic is produced, which is noticed if the charcoal is removed from the flame and smelt. The flame is also coloured *livid blue.*

A little solid As_2O_3 heated in a small ignition tube is readily "sublimed," and the sublimate is seen under a microscope or a powerful lens to consist of octahedral crystals.

B. DISTINCTIVE TESTS FOR ARSEN*IOUS* AND ARSEN*IC* COMPOUNDS.

Reagent.	Arsen*ious* compound. Use As_2O_3 dissolved in HCl except for (187).	Arsen*ic* compound. Use $Na_2HAsO_4.12H_2O$ dissolved in H_2O.
187. *$AgNO_3$*, several drops are added to a solution of As_2O_3 in water. For this reaction As_2O_3 dissolved in boiling water is employed, because HCl would yield a precipitate of AgCl.	*Light-yellow precipitate*, (Ag_2HAsO_3 or Ag_3AsO_3): this precipitate forms only on adding carefully, drop by drop, a little dilute AmHO*: show that it is dissolved by AmHO and by HNO_3. It is also appreciably soluble in $AmNO_3$: hence, if too much AmHO has been added, the precipitate does not always form on neutralizing with HNO_3.	*Brown precipitate*, (Ag_3AsO_4): show that it is soluble in AmHO and in HNO_3.
188. *$CuSO_4$*, several drops:	*Yellowish-green precipitate* ($Cu''HAsO_3$): this precipitate forms only on adding dilute AmHO carefully, drop by drop†: show that it is soluble in AmHO and in HNO_3.	*Pale-green precipitate*, ($CuHAsO_4$): soluble in AmHO and in HNO_3.
189. *$CuSO_4$*, two drops: then KHO added:	The above yellowish-green precipitate is first produced, but on addition of more KHO this dissolves to a clear *blue* liquid which on being boiled deposits *red* Cu_2O.	No clear blue solution is obtained, and no Cu_2O is produced on boiling the liquid.
190. *AmCl*, *AmHO*, and *$MgSO_4$*:	No precipitate.	White crystalline precipitate($MgAmAsO_4$): if filtered off and moistened with $AgNO_3$ solution on the filter it becomes *brown*.

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* As_2O_3 when dissolved in water yields H_3AsO_3 which cannot give with $AgNO_3$ the Ag_3AsO_3 precipitate because this decomposition would liberate HNO_3 in which Ag_3AsO_3 is soluble; hence the addition of AmHO is necessary to neutralise the free acid; but since the precipitate is also soluble in AmHO, the latter must be added carefully in only just sufficient quantity to neutralise the acid. This is best effected by using some very dilute AmHO, made by pouring a few drops into a test-tube, filling the tube two-thirds with distilled water, then closing it with the thumb, and inverting it for a moment. The dilute AmHO is to be added drop by drop, shaking or stirring the test solution after each addition until the precipitate is obtained.

† The precipitate produced by $CuSO_4$ is soluble in acids, hence the HCl present in the solution must be neutralised by careful addition of AmHO before the precipitate will appear.

ANTIMONY (Sb).—Use $SbCl_3$, or $2KSbO\overline{T}.H_2O$ "Tartar emetic," dissolved in dilute HCl.

Antimon*ic* compounds give results ultimately identical with antimon*ious* in the following eight reactions, except in (192). After these reactions, which are common to Sb in both its sets of compounds, are given two which are distinctive: they are, however, rarely used and may be neglected by the student.

191. *H_2S:* orange-red precipitate (Sb_2S_3): pour into two tubes and show that the precipitate is dissolved when warmed with pure NaHO or with Am_2S, but is reprecipitated from these solutions on addition of HCl in excess.

192. If poured into much water, $SbCl_3$ solution yields a white precipitate or turbidity (SbOCl): this precipitate redissolves on adding HCl and warming, and is also readily soluble in $H_2\overline{T}$; in this latter respect it differs from the similarly formed BiOCl precipitate (156).

193. If a few drops of acid $SbCl_3$ solution be poured upon a piece of platinum foil* and a piece of Zn be dropped into it, a black deposit of Sb will rapidly form *on the foil.* If the foil be rinsed with water, and then boiled with HCl, the stain remains undissolved, but it is rapidly removed by boiling HNO_3.

TESTS DEPENDING ON THE FORMATION OF SbH_3:—

SbH_3 differs from AsH_3 in not being poisonous, and in being free from smell.

194. If SbH_3 gas, formed by the action of acid $SbCl_3$ solution on Zn, is passed into $AgNO_3$ solution in a precisely similar way to that described for AsH_3 (181), a black precipitate ($SbAg_3$) forms:—

$$SbH_3 + 3AgNO_3 = SbAg_3 + 3HNO_3.$$

This precipitate is to be filtered off, and the filtrate, which will contain no Sb, rejected. Wash the precipitate four or five times with boiling water upon the filter: then pour upon it boiling dilute $H_2\overline{T}$ solution, which will dissolve the Sb, and receive the liquid in a test-tube; boil it, and pour it once more upon the filter; add HCl to the liquid, filter if necessary, and pass H_2S into it, orange-red Sb_2S_3 will be precipitated.

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* A slip of platinum foil laid in a watch glass or porcelain dish, or with turned up edges, may be employed: the inverted lid of a platinum crucible *is very convenient* for this purpose.

195. A stain produced by Sb on porcelain, in the manner already described (182), differs from the As stain in presenting a *dead* surface and in being insoluble in Na_2Cl_2O; also when the stain is dissolved by warming with Am_2S, the solution if gently evaporated leaves *orange-red* Sb_2S_3.

196. The Sb mirror, obtained as described in (183), differs from the As mirror by being formed much nearer to the flame, and on both sides of the heated part of the tube; it may further be distinguished by the tests given in (195). This mirror, after oxidation, is also insoluble in boiling water: and if dissolved in a little boiling HCl the solution gives an orange-red precipitate (Sb_2S_3) when H_2S is passed into it, whereas the As_2O_3 solution gives yellow As_2S_3.

196 *a.* SbH_3 if allowed to act on filter-paper moistened with $HgCl_2$ solution, as described in 183 *a* for AsH_3, yields a *greyish-brown* stain.

197. Boiled with Zn or Al and KHO, no SbH_3 is evolved. [Difference from As (184)].

198. If a little solid $KSbO\overline{T}$ or Sb_2O_3, or any substance containing Sb, be mixed in a cavity on wood charcoal with Na_2CO_3 and KCy, and the mixture be heated in the inner blowpipe flame, a white incrustation forms on the charcoal, and white globules of metallic Sb are obtained which are extremely brittle. The flame is coloured pale green. If the melted Sb be allowed to stand aside on the charcoal, the globule emits a white smoke, and coats itself with sharp white crystals of the oxide.

The two distinctive tests (199, 200) for antimonious and antimonic compounds given in the following Table are seldom used; they may be read through without trying the reactions, and may be referred to hereafter if required for analytical purposes.

DISTINCTIVE TESTS FOR ANTIMON*IOUS* AND ANTIMON*IC* COMPOUNDS.

Reagent.	**Antimon*ious*.** (Use the above solution of $SbCl_3$ in dilute HCl.)	**Antimon*ic*.** (Use $K_2Sb_2O_7$ solution, obtained by boiling some "Potassium metantimonate" with H_2O).
199. Add excess of KHO, then $AgNO_3$ solution:	A dark-coloured precipitate which when shaken after addition of AmHO leaves black Ag_4O undissolved.	A brown precipitate, which dissolves entirely on addition of AmHO.
200. Add excess of HCl and warm; then pour in a little KI solution:	No iodine is set free; proved by the liquid not turning brown and giving no colour after being cooled and mixed with freshly-prepared starch solution.	Iodine is liberated, giving a brown colour to the liquid and yielding a deep blue colour when starch solution is added to the cold liquid.

TIN (Sn).—Stannosum and Stannicum.

DISTINCTIVE REACTIONS FOR STANN*OUS* AND STANN*IC* COMPOUNDS.

Test.	Stann*ous* Compounds. Use $SnCl_2.2H_2O$ dissolved in dilute HCl.	Stann*ic* Compounds. Use $SnCl_4$ in dilute HCl.
201. Pass H_2S:	*Dark-brown precipitate* (SnS): pour off two portions and show that the precipitate is soluble in KHO and in *yellow* Am_2S on heating; and is precipitated again by HCl, from the KHO solution as brown SnS, from the Am_2S solution as yellow SnS_2. SnS differs from SnS_2 in being insoluble in *colourless* Am_2S, which readily dissolves SnS_2.	*Yellow precipitate* (SnS_2), often forming only when the liquid is heated: pour off portions and show that the precipitate is soluble in Am_2S (both *yellow* and colourless) and in KHO on heating, and is precipitated again by HCl as yellow SnS_2 from both solutions.
202. $HgCl_2$:	*A white precipitate* (Hg_2Cl_2): turns grey on being boiled if the $SnCl_2$ is in excess (145).	——
203. $AuCl_3$: added after a few drops of $SnCl_4$ solution or of Cl water:	*Purple* or *dark brown precipitate*, "Purple of Cassius."	——

Reactions common to Stannous and Stannic compounds.

204. *Zn* in presence of HCl precipitates from Sn solutions a spongy mass of Sn : the Zn and solution should be allowed to stand together for some time in a small porcelain dish, the action is much hastened by gentle heat. If this test is done on platinum foil the Sn is deposited in a spongy state *on the Zn, and does not stain the Pt* (difference from Sb). $SnCl_4$ is not so readily acted upon by Zn as $SnCl_2$ is; from $SnCl_2$ the Sn is often precipitated in beautiful crystals.

205. A solid Sn-compound, $SnCl_2$ or SnO_2, mixed with powdered Na_2CO_3 and KCy and heated on charcoal in the inner blowpipe flame, gives a slight white incrustation and white particles of metallic Sn which are with difficulty fused into globules. The globules of Sn differ from those of Pb by not marking paper (153).

206. GROUP II.B.—TABLE OF DIFFERENCES.

The following tests answer for these elements in both sets of compounds : for distinctive tests see the preceding reactions.

Tests.	As.	Sb.	Sn.
a. For liquids.			
1. Pass H_2S into the solution acidified with HCl and heat; if no precipitate forms heat to boiling, and again pass H_2S:	*Yellow* precipitate, insoluble in boiling strong HCl.	*Orange-red* precipitate, soluble in boiling strong HCl.	SnS — *Brown* precipitate. SnS_2 — *Yellow* precipitate. Both soluble in boiling strong HCl.
2. Zn and HCl:	*AsH_3* gas is evolved, which if passed into $AgNO_3$solution gives a black precipitate of Ag, H_3AsO_3 being left in solution; on adding *dilute* AmHO to this solution, yellow Ag_3AsO_3 precipitates.	*SbH_3* gas is evolved, which if passed into $AgNO_3$ solution gives a precipitate of Ag_3Sb, from which hot $H_2\bar{T}$ solution dissolves Sb; H_2S gives in this solution after adding HCl orange-red Sb_2S_3.	*Sn* is deposited on the Zn strips.
3. Zn and HCl on platinum:	AsH_3 evolved, *no stain on the platinum.*	*Black stain of Sb on the platinum.*	Sn deposited on the zinc, *no stain on the platinum.*
b. For solids.			
4. Heated with Na_2CO_3 and KCy on charcoal in the inner blowpipe flame:	*No* metallic globules; smell of garlic.	*Brittle* metallic globules, white incrustation.	*Malleable* metallic globules, white incrustation.
5. Heated with solid Na_2CO_3 and KCy in a bulb tube:	Black mirror and smell of garlic.	——	——

The detection of any one member of this group is simple; the colour of the precipitate with H_2S, confirmed by special tests, is quite sufficient to identify it with ease.

207. (S) *Many methods have been proposed for the separation and detection of As, Sb, and Sn, when mixed;* two of the most trustworthy are those introduced by *Hofmann* (208) and by *Fresenius* (210); a method more simple, though less trustworthy, is given in (209).

208. (s) *Hofmann's* method consists in pouring the solution into a flask, in which H is being generated by the action of HCl upon Zn; Sn remains as a spongy mass on the Zn, As and Sb are evolved as AsH_3 and SbH_3; these gases, if passed into $AgNO_3$ solution, yield soluble H_3AsO_3 and insoluble Ag_3Sb, which are separated by filtration; the three members of this group thus separated are then detected by special tests.

As, Sb, Sn, occurring together in a solution, are tested for by precipitating them with H_2S, filtering, and testing the filtrate by passing H_2S through it as was directed for precipitating Group II.A. The precipitate is then examined by Table II. (435 *b*, Arsenic Group), using only the right hand side of the Table.

The H_2S precipitate may also be examined by the simpler method given in (209), or by the very accurate, but less simple method in (210).

209. (s) The following method of examining a precipitate, produced by H_2S, for As, Sb, Sn is recommended by its comparative simplicity; it is sufficiently accurate for ordinary analyses where traces of these metals have not to be looked for. It depends upon the insolubility of As_2S_3 in hot strong HCl, in which SnS, SnS_2, and Sb_2S_3 are soluble; the further separation of Sb and Sn is effected either by adding Zn alone to the acid solution, when Sb is evolved as SbH_3 and Sn is deposited upon the Zn, or by adding Zn and Pt, when Sn is deposited on the Zn and Sb on the Pt.

The precipitate produced by H_2S is filtered off and allowed to drain for some time upon the filter in the funnel; it is best to drain it still further by carefully taking the filter out of the funnel, opening it out, and spreading it upon a piece of filter-paper folded several times upon itself; the precipitate is then removed to a small porcelain dish and heated for some time nearly to boiling with strong fuming HCl; the liquid is cooled and filtered:—

The residue will consist chiefly of As_2S_3, which is almost insoluble in strong HCl.

Dry the washed residue on the filter at a gentle heat, then mix it with three or four times as much powdered KCy and Na_2CO_3, and heat the mixture in a small bulb-tube, removing any drops of water inside the tube by a twisted piece of filter-paper; a black mirror (185):—*Presence of As.*

Note.—The As_2S_3 may also be dissolved by heating with a little fuming HNO_3. The excess of HNO_3 is boiled off and As detected as H_3AsO_4 by adding AmCl excess of AmHO and $MgSO_4$; or by addition of $AgNO_3$, and then cautiously neutralising with AmHO.

The filtrate may be examined for Sb and Sn by either I. or II. below:—

I. Place a piece of platinum-foil in a porcelain dish and pour the acid filtrate upon it, then touch the foil with a piece of Zn; H will come off with effervescence, and if either at once or after a few minutes a black stain appears upon the platinum the *presence of Sb* is indicated*.

The platinum-foil is removed and pieces of Zn are placed in the liquid: as soon as the bubbles of H cease to be given off, Zn still remaining undissolved, remove the pieces of Zn, rubbing and rinsing any dark deposit back into the dish; let this deposit settle, decant the liquid and heat the solid deposit with strong HCl for several minutes in a test-tube, dilute with a little water, filter if necessary, and add to the solution a few drops of $HgCl_2$ solution: a white or grey precipitate (204, 202) indicates the *presence of Sn.*

II. The acid filtrate is poured into a little hydrogen flask (fig. 34, p. 114), in which H has been coming off briskly for about five minutes, being produced by the action of a little strong HCl upon some pieces of Zn. The H is lighted at the jet, and the inside of a small porcelain dish or crucible lid pressed down upon the flame: a black stain not dissolved by hot Na_2Cl_2O solution shows the *presence of Sb.*

The residue in the flask is tested for Sn, as directed in the latter part of I. (above).

210. (s) The following method of detecting As, Sb, Sn, described by Fresenius, is very delicate and trustworthy. It depends upon the fact that fusion with Na_2CO_3 and $NaNO_3$ converts As_2S_3 into soluble Na_2HAsO_4, Sb_2S_3 into insoluble $Na_2H_2Sb_2O_7$, and SnS_2 into insoluble Sn or SnO_2.

The precipitated sulphides are dried on the filter, and the precipitate† mixed well on a watch glass or in an agate-mortar with equal weights of powdered Na_2CO_3 and $NaNO_3$; this powder is dropped in small quantities at a time into some $NaNO_3$ kept melted in a small porcelain crucible over a Bunsen flame. When the powder has all been added, the crucible is heated sufficiently to keep the substance melted for a few minutes, and the melted mass is then poured into a small dry porcelain dish; as soon as the substance has cooled, cold water is poured upon it in the dish and also upon the residue adhering to the inside of the crucible, and the solid substance is allowed to soak for some time with occasional stirring, crushing it by pressure with a pestle if it does not easily fall to pieces; it is then filtered:—

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* The Sb thus deposited on the Pt may, after rinsing the foil, be dissolved by heating the Pt in a test-tube with a little very dilute HNO_3; on cooling, diluting, and passing H_2S, an orange-red precipitate will form, confirming the *presence of Sb.*

† *If the* quantity of the precipitate is very small, the part of the paper

The residue is washed on the filter with a mixture of equal measures of water and alcohol, to prevent solution of the $Na_2H_2Sb_2O_7$, the washings are rejected. The precipitate is then rinsed into a small porcelain dish, using as little water as possible; a few drops of strong HCl are added and the dish is heated, more HCl being added, if necessary, to make the liquid acid to litmus-paper. Whether the residue has dissolved or not, place in the liquid a piece of clean platinum-foil, and upon the foil a piece of pure Zn. If a black stain forms after a time upon the platinum, it proves the *presence of Sb**. When the Zn ceases to be acted upon, Sn if present will remain as a spongy deposit. This is to be washed by decantation, dissolved by boiling with HCl in a test-tube, and $HgCl_2$ added immediately to the solution; a white precipitate (202) shows the *presence of Sn*.	*Filtrate:* HNO_3 is added until the solution is just acid after being boiled, then $AgNO_3$ solution is poured in as long as it causes any precipitate, and very dilute AmHO is added little by little †; a brown precipitate, readily dissolving in excess of AmHO, shows the *presence of As*.

GROUP I.—SILVER GROUP.

211. This group includes Pb, Ag, Hg′ (Mercurosum); its members differ from those of all the other analytical groups by being precipitated as chlorides by HCl: since, however,

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containing the precipitate is cut up into small pieces and treated as the precipitate.

* See the first foot-note on p. 124.

† Instead of mixing dilute AmHO with the liquid, it may be carefully poured upon the top of the liquid; a brown colour is then seen at the surface of contact.

$PbCl_2$ is rather soluble in water, Pb is not entirely precipitated by HCl, and is usually found also in Group II.A. as PbS.

LEAD (Pb).—Reactions already given in Group II.A. (149–153).

SILVER (Ag).—Use $AgNO_3$ solution.

212. *HCl:* white precipitate (AgCl), becomes curdy on being shaken or heated: pour off into four tubes; filter one, wash the precipitate and let it stand for some time exposed to sunlight or common daylight, it will become blackish-purple; show with the other portions that AgCl dissolves readily when heated with AmHO or with KCy solution, and is precipitated again from these solutions on adding HNO_3 in excess; also that it is insoluble in HNO_3 even on boiling.

213. *H_2S or Am_2S:* black precipitate (Ag_2S), insoluble in Am_2S or KHO, soluble in boiling dilute HNO_3.

214. *KHO:* brown precipitate (AgHO), insoluble in excess.

215. *AmHO:* light-coloured precipitate, produced only when *very dilute* AmHO is added drop by drop, easily soluble in excess of AmHO.

216. A mixture of a solid Ag-compound and powdered Na_2CO_3 heated on charcoal, gives white malleable globules or scales of Ag and no incrustation.

MERCUROSUM (Hg′).—Use $Hg'_2(NO_3)_2.H_2O$ dissolved in dilute HNO_3.

Reactions (145, 146, 147, and 148) given under Mercuricum (Hg″) yield precisely similar results with Mercurosum (Hg′) and may be repeated with $Hg'_2(NO_3)_2$ solution.

In reaction (146) however it is not necessary to add acid since the $Hg'_2(NO_3)_2$ solution is already acid.

217. *HCl:* a white precipitate (Hg'_2Cl_2), insoluble in dilute acids unless warmed with both HCl and HNO_3, which convert it into soluble $Hg''Cl_2$: *becomes black when AmHO is poured upon it, but does not dissolve.*

218. *AmHO or KHO*: black precipitate, insoluble in excess.

219. *H_2S*: black precipitate (Hg'_2S): this precipitate like $Hg''S$ (144) is insoluble in Am_2S, in hot strong HCl, and in hot strong HNO_3, but is easily dissolved when heated with a mixture of HNO_3 and HCl.

220. GROUP I.—TABLE OF DIFFERENCES.

Tests.	Pb.	Ag.	Hg′.
a. For liquids.			
1. Add HCl:	White precipitate, *soluble in boiling water; AmHO neither dissolves the precipitate nor changes its colour.*	White curdy precipitate, *insoluble in boiling water, easily soluble in warm AmHO,* reprecipitated from this solution by adding HNO_3 in excess.	White precipitate, *insoluble in boiling water, and insoluble in AmHO, but blackened by it.*
2. Add AmHO:	*White precipitate,* insoluble in excess.	*Brown precipitate,* readily soluble in excess.	*Black precipitate,* insoluble in excess.
3. Add K_2CrO_4:	*Bright yellow* precipitate.	*Chocolate-red* precipitate.	*Scarlet-red* precipitate.
b. For solids.			
4. Fused with Na_2CO_3 on charcoal in the inner blowpipe flame:	*White malleable globules of metal* which mark paper; yellow incrustation on the charcoal.	*White malleable globules or scales* which do not mark paper; no incrustation.	No metallic globules.
5. Heated in a bulb-tube, mixed with Na_2CO_3:	——	——	*Grey sublimate,* consisting of globules of metallic mercury.

By the above differences any one member of this group is readily distinguished.

221. (s) *The separation and detection of Pb, Ag, and Hg′, when mixed,* depend upon :—

1st. The solubility of $PbCl_2$ in boiling water, in which AgCl and Hg'_2Cl_2 are insoluble.

2nd. The solubility of AgCl in AmHO, in which Hg'_2Cl_2 is insoluble.

The presence of each member, when thus separated is then confirmed by one of its special tests.

222. (s) A solution which is to be examined for Pb, Ag, Hg′ is precipitated by HCl added in excess, then filtered, and the clear filtrate is tested with more HCl to ascertain that no further precipitate is produced. The precipitate is then examined by Table I. (433.)

The foregoing reactions only include those for the metals of common occurrence. For information concerning the detection and separation of the rarer metals, which will only be required by a somewhat advanced student, the appendix must be consulted. The metals gold and platinum, however, are briefly noticed here on account of their frequent employment for chemical and other purposes. These two metals are dissolved only by a mixture of HCl and HNO_3: their most important reactions are given below.

GOLD (Au).—Use $AuCl_3$ solution.

Gold solutions are usually bright yellow in colour.

223. *$SnCl_2$* containing a little $SnCl_4$: purple or dark-brown precipitate ("Purple of Cassius"), best seen by performing the experiment in a white porcelain dish.

224. *$FeSO_4$* produces either at once, or on heating the solution, a very finely-divided precipitate of Au: the liquid usually appears bluish by transmitted light; always copper-red by reflected light:—

$$2AuCl_3 + 6FeSO_4 = 2Au + 2Fe_2(SO_4)_3 + Fe_2Cl_6.$$

224 *a.* *H_2SO_3* produces on boiling a similar precipitate of Au: by boiling the liquid for some time in a porcelain dish, the Au settles in small black masses and the solution loses its colour.

PLATINUM (Pt).—Use $PtCl_4$ solution.

225. *AmCl* added to a strong solution produces on standing for some time, or more quickly on being stirred, a yellow precipitate (Am_2PtCl_6): rather soluble in hot water.

226. *$SnCl_2$* gives a reddish-brown colour in the solution acidified with HCl, owing to the formation of $PtCl_2$:—

$$PtCl_4 + SnCl_2 = PtCl_2 + SnCl_4.$$

The method of separating and detecting Au and Pt is fully stated in paragraphs 472–474.

⁂ *Note.*—Before commencing the reactions for acid-radicles the student may with advantage analyse several substances which are liable to contain any one or more of the members of the metallic groups. If only *one* metal has to be looked for (see column 1, paragraph 539), the directions given in paragraphs (330–369) may be followed, omitting those portions which relate to the detection of acid-radicles. If two or more metals may be present (see columns 2, 3, paragraph 539), full directions for ascertaining to which analytical group or groups they belong will be found in the General Table (419); and the group precipitate or precipitates thus obtained may be then examined by the Group Tables (433–439), with which the student will probably already be familiar.

REACTIONS FOR ACID-RADICLES.

In the course of analysis acid-radicles are usually detected by special tests: they cannot advantageously be precipitated in groups and the members of each group then separated and identified, as is done in the case of metals. Accordingly the arrangement here adopted consists in simply placing together in a group such acid-radicles as in some respects resemble one another in their reactions, and at the end of each group stating upon what differences the detection of its members when occurring together depends.

The reactions for acid-radicles are worked through in a manner similar to that employed for the reactions of the metals,—a compound (usually a salt) containing the radicle being employed. At the end of each group the student may with advantage detect one or more of its members, as was done with the metals.

GROUP I.—SULPHATE GROUP.

Sulphates are the only commonly occurring salts which give with $BaCl_2$ a precipitate insoluble in boiling HCl. Fluosilicates resemble sulphates in this respect, but differ in so many other reactions that they are considered hereafter (299–302) in connection with fluorides and silicates, to which they are more closely related.

SULPHATES (- ″SO_4).—Use $Na_2SO_4.10H_2O$.

227. *$BaCl_2$ or $Ba(NO_3)_2$*: white precipitate ($BaSO_4$), insoluble when boiled with dilute HCl or HNO_3.

If $BaCl_2$ or $Ba(NO_3)_2$ be added to a solution containing much strong HCl or HNO_3, a white precipitate may be produced consisting of $BaCl_2$ or $Ba(NO_3)_2$, which however is readily distinguished from the precipitate of $BaSO_4$ by being dissolved when boiled with water.

$Sr(NO_3)_2$ and *$Pb(NO_3)_2$* give also white precipitates ($SrSO_4$ and $PbSO_4$).

228. *Blowpipe test.*—Solid $Na_2SO_4.10H_2O$ or any solid sulphate, if mixed with solid Na_2CO_3* and fused on charcoal in the inner blowpipe flame *produced from a spirit-flame*, yields Na_2S :—

$$Na_2SO_4 + 4C = Na_2S + 4CO.$$

The Na_2S is readily detected by detaching the solid mass, after it has cooled, from the charcoal with the point of a knife; placing a portion of it upon a bright silver coin, then moistening it with a drop of water and crushing it by pressure with a knife-blade. After the moistened mass has remained upon the coin for a short time rinse it off, a black stain will then be seen upon the silver where the substance had rested (231).

Another portion of the cool mass may be moistened with a drop of HCl, when the fetid smell of H_2S will be observed, and a piece of paper moistened with $Pb\bar{A}_2$ solution and held above it, will be blackened (230).

This test for a sulphate is reliable only when it is known that sulphur in no other form is present in the substance to be tested; it is also necessary that no sulphur should be introduced in the Na_2CO_3, or by the flame. Now coal-gas sometimes contains sufficient sulphur to give the above reaction with pure Na_2CO_3, hence it is always advisable to employ a spirit- rather than a gas- flame.

Hydrogen sulphate, or sulphuric acid (H_2SO_4), may be detected by producing black charred stains on paper which has been dipped into it and dried by heat: it also has a strong acid reaction to litmus, and evolves hydrogen with Zn, properties shown however by some metallic and acid sulphates.

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*Addition of $Na_2B_4O_7$ is useful in preventing the fused mixture from sinking into the charcoal.

GROUP II.—THE CARBONATE GROUP.

The five following classes of salts give off characteristic gases when acted upon by HCl.

⁂ The systematic Tables of Differences, such as are given for each group of the metals, are not drawn out for the acid-radicle groups; the student should have no difficulty in constructing such tables for himself, either mentally or on paper, by looking through the reactions.

CARBONATES ($-''CO_3$).—Use Na_2CO_3, or white marble ($CaCO_3$).

229. *HCl* or $H\bar{A}$ or almost any other acid, if poured upon some Na_2CO_3 in a test-tube, causes carbon-dioxide gas (CO_2) to come off with effervescence: this gas is recognised by its property of turning lime- or baryta- water milky by the production in them of insoluble $CaCO_3$ or $BaCO_3$.

The test may be tried in several ways:—

1. The acid is poured upon the carbonate in a test-tube. A glass rod which has been dipped into some perfectly clear lime-water * and has a small drop adhering to its end is then introduced into the test-tube, carefully avoiding touching the liquid or the sides of the tube; the drop will quickly become milky.

2. The acid is poured upon the Na_2CO_3 in a small beaker, and this is immediately covered with a watch glass placed with its convex face downwards, having a drop of clear lime-water * adhering to the middle of the face: the drop becomes milky.

3. Since CO_2 gas is much heavier than air, on adding acid the gas evolved from the carbonate will remain in the test-tube if the tube is held erect and its mouth loosely closed with the thumb: by gradually sloping the tube the heavy gas is poured into another test-tube containing lime-water * without allowing any liquid to flow out, on closing this tube with the thumb and well shaking it the lime-water will

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* Baryta-water gives a much more decided result than lime-water.

become milky: or if the inside of the tube has been rinsed with lime-water the sides become white.

FIG. 37.

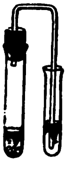
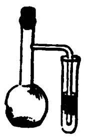


FIG. 38.



4. The CO_2, if it is given off in considerable quantity, may also be made to pass into some lime-water through a doubly bent tube fitted air-tight by a perforated cork or india-rubber stopper into the tube in which the CO_2 is generated, as shown in the fig. 37; or the apparatus shown in fig. 38 may be employed.

Of the above methods 1 and 3 are the most simple; by method 4 the CO_2 is not found unless given off in somewhat large quantity.

The following precautions must be attended to. Since the milkiness at first caused by CO_2 disappears gradually when more CO_2 is absorbed, if the lime-water does not at once become milky, it must be constantly watched to avoid the risk of its becoming milky and again clear before being seen.

Also if the addition of the first few drops of acid does not cause an effervescence of CO_2 more acid should be added, as many carbonates retain the first portions of CO_2 by forming acid carbonates:—

$$Na_2CO_3 + CO_2 + H_2O = 2NaHCO_3.$$

A solid substance which is being tested by HCl for CO_2 should be first moistened so as to drive out the air-bubbles entangled in it, which, in coming off, might make one suspect effervescence of CO_2.

Hydrogen carbonate, or *carbonic acid* (H_2CO_3), can only exist in dilute aqueous solution; addition of lime-water to its solution causes milkiness; but, since other substances in solution behave in a similar way, it is best found by boiling the liquid and passing the steam with the CO_2 which accompanies it into lime-water; the lime-water becomes milky.

SULPHIDES (– ″S).—Use FeS for solid, and Am_2S for liquid.

230. *HCl or H_2SO_4* poured upon a small fragment of FeS causes H_2S gas to come off with effervescence: this gas is detected by its fetid smell, resembling that of rotten eggs, and by its property of blackening $Pb\bar{A}_2$-solution by the formation of PbS. The gas may be made to act upon the $Pb\bar{A}_2$ solution by the methods given for CO_2 (229), or better by placing a piece of filter-paper moistened with $Pb\bar{A}_2$-solution on the end of a glass rod so that one-half of the slip adheres to the glass rod and the other hangs free, and holding the rod in the gas as in the test for CO_2 (229, 1).

This test is made much more delicate if the paper is moistened with a solution prepared by adding KHO to boiling $Pb\bar{A}_2$-solution until the precipitate first formed dissolves: a test-paper thus prepared becomes intensely blackened by H_2S.

231. Solutions of soluble sulphides if dropped upon a bright silver coin produce a black stain (Ag_2S); this may be removed by rubbing the coin with moist lime.

232. Insoluble sulphides which give off H_2S with HCl (*e.g.* FeS) produce a black stain when placed on a silver coin and moistened with a drop of HCl.

233. *$Pb\bar{A}_2$*-solution gives with soluble sulphides a black precipitate (PbS). The solution produced by adding KHO in excess to $Pb\bar{A}_2$-solution is more delicate than $Pb\bar{A}_2$ alone.

234. Many sulphides (*e.g.* FeS_2, "Iron Pyrites") and also free S, if strongly heated in a tube open at both ends and held obliquely in the flame, or in the tube shown in fig. 35 (p. 114), give off SO_2 gas, recognised by its pungent smell and by turning paper moistened with $K_2Cr_2O_7$ green.

235. Reaction (306) given under HCy may be employed also as a very delicate test for soluble sulphides. Test (228) for a sulphate, also answers for a sulphide.

Hydrogen sulphide, or hydrosulphuric acid (H_2S), in aqueous solution is easily recognised by (231) or (233), and unless the solution is very dilute, also by its smell, and by suspending lead-paper over the liquid: H_2S gas is detected by (230).

SULPHITES ($-''SO_3$).—Use Na_2SO_3.

236. *HCl or H_2SO_4* poured upon Na_2SO_3 and warmed gives off SO_2 gas, known by its pungent smell and by turning $K_2Cr_2O_7$ solution green: the $K_2Cr_2O_7$ solution may be exposed to the gas by the methods described under (229), preferably by introducing a glass rod with a drop hanging upon its end into the gas; or by moistening a small strip of filter-paper with the $K_2Cr_2O_7$ solution and making it adhere by one-half of its length to the rod (230), the other half hanging freely, and introducing it into the gas as in (229, 1).

237. *BaCl*$_2$: white precipitate ($BaSO_3$): entirely soluble in HCl, unless some Na_2SO_4 is present, when $BaSO_4$ remains undissolved: on adding Cl- or Br-water to the HCl solution, $BaSO_4$ is formed and precipitated:—

$$BaSO_3 + 2HCl = BaCl_2 + H_2SO_3.$$
$$BaCl_2 + H_2SO_3 + H_2O + 2Cl = BaSO_4 + 4HCl.$$

238. *Na*$_2$*SO*$_3$ added to a mixture of *HCl and Zn* which is giving off pure H, immediately causes an evolution of H_2S with the H: the H_2S is detected by its smell, or by blackening a piece of filter-paper moistened with solution of PbO in excess of KHO (230).

Hydrogen sulphite or *sulphurous acid* (H_2SO_3), can be found in aqueous solution by adding a drop of $K_2Cr_2O_7$ solution, which will at once become green: SO_2 gas is found as directed under (236).

THEIOSULPHATES* ($-''S_2O_3$).—Use $Na_2S_2O_3.5H_2O$.

239. *HCl or H*$_2$*SO*$_4$ causes no immediate change in the cold, but the liquid on standing or on being warmed deposits a precipitate of yellow S, and SO_2 is given off; the SO_2 is recognised by its pungent smell and by changing the colour of $K_2Cr_2O_7$ solution (236).

240. *Fe*$_2$*Cl*$_6$: gives a reddish-violet colour which vanishes after a short time or immediately when heated, the Fe_2Cl_6 solution at the same time loses its yellow colour owing to the change of Fe_2Cl_6 into $FeCl_2$.

240 *a.* *AgNO*$_3$; a white precipitate ($Ag_2S_2O_3$), becoming black after standing for a short time, or immediately upon being heated, owing to the formation of Ag_2S:—

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* Formerly known as hyposulphites.

$$Ag_2S_2O_3 + H_2O = Ag_2S + H_2SO_4.$$

The above precipitate ($Ag_2S_2O_3$) dissolves very readily in excess of the $Na_2S_2O_3$ solution, hence it is most easily obtained by dropping the latter into some $AgNO_3$ solution.

Hydrogen theiosulphate, or *theiosulphuric acid* ($H_2S_2O_3$), is extremely unstable, rapidly separating into $S + H_2SO_3$.

HYPOCHLORITES ($-'ClO$).—Use $NaClO.NaCl$ or Na_2Cl_2O solution.

Hypochlorites give off a faint smell of Cl in the air if solid or in strong solution.

241. *HCl or* H_2SO_4 *or even* $H_2\bar{O}$ in the cold, or when gently warmed if much water is present, sets free Cl-gas, which may be recognised by its smell, its yellowish colour, and by bleaching a piece of moistened litmus-paper, which is held for a short time in the tube without touching the liquid or the sides of the tube.

A piece of litmus-paper shaken up with the solution, without addition of acid, is also bleached, the CO_2 in the air probably liberating the Cl: on addition of a drop of any acid the bleaching is very rapid:—

$$Na_2Cl_2O + 2HCl = 2Cl + 2NaCl + H_2O.$$

242. $Pb(NO_3)_2$ *or* $Pb\bar{A}_2$ solution added in large quantity gives a white precipitate, becoming reddish, and then dark brown (PbO_2) when boiled for a short time.

$MnCl_2$ likewise gives a *dark brown* precipitate on heating.

Hydrogen hypochlorite, or *hypochlorous acid* ($HClO$), is a yellow liquid with sweetish smell, which if strong is very unstable: it bleaches litmus, and evolves Cl when warmed with HCl. Cl_2O gas is of a deep yellowish-green colour, and has an irritating smell, it dissolves easily in water to form the above acid.

Nitrites ($-'NO_2$).—Use KNO_2.

243. Warmed with dilute H_2SO_4, reddish-brown "nitrous fumes," with a characteristic smell, are given off; they are best seen by looking down the tube at some white object.

244. If cold $FeSO_4$ solution is added to KNO_2 solution, it becomes brown; on adding cold dilute H_2SO_4 the colour becomes much more intense: it is produced by the combination of NO with $FeSO_4$: this colour is destroyed by boiling, red fumes being given off (243).

245. If solution of KI and several drops of starch solution (par. 526, remark 35) are added to KNO_2 solution, and the liquid is then made acid with $H\bar{A}$, an intense blue colour will be produced, owing to the combination of iodine, set free by the HNO_2, with the starch; the experiment is best done in a white procelain dish, the liquid, which often appears black at first, being diluted, if necessary, till its blue colour becomes visible. This is a most delicate test, and is rather characteristic if the colour is not produced till the $H\bar{A}$ is added.

Hydrogen nitrite, or *nitrous acid* (HNO_2) is extremely unstable, decomposing into HNO_3, NO, and H_2O.

⁂ The student will have no difficulty in detecting any one of the above acid-radicles occurring singly in a liquid or solid: warming with HCl will usually indicate which of them is present, and its presence may then be confirmed by a second test.

246. (s) Most of the acids formed by the salts of Group II. on addition of HCl, react upon one another; *e.g.* HClO destroys HNO_2, H_2S, and H_2SO_3; and H_2S destroys H_2SO_3. The analyst may often by skilful use of the above reactions detect these acids when mixed, but their detection in certain mixtures is a problem only soluble by careful consideration, and not unfrequently insoluble.

SO_2, if evolved, may be detected by $K_2Cr_2O_7$ paper: H_2S by $Pb\bar{A}_2$ paper: Cl by bleaching moist litmus-paper: N_2O_3 by its brown colour. But CO_2 can only be detected in presence of much SO_2 by passing the gases through hot $K_2Cr_2O_7$ solution which absorbs SO_2, CO_2 passing on and being detected by lime-water.

GROUP III.—NITRATE GROUP.

The two classes of salts which follow resemble one another in many respects, more particularly in the fact that no member of them is insoluble in water; hence no method of precipitating them is known.

NITRATES ($-'NO_3$).—Use KNO_3.

247. Add to some KNO_3 solution contained in a rather broad test-tube about an equal bulk of strong H_2SO_4; cool the liquid by letting a stream of cold water run upon the tube from the tap; then hold the tube in a slanting position, and pour some strong cold $FeSO_4$ solution in a gentle stream down the inside of the tube. If this solution is poured in with proper care it will form a distinct layer resting on the surface of the acid liquid, and at the surface of contact of the two layers a *dark brown* film or ring will appear, sometimes extending upwards towards the surface of the liquid. The colour is most easily seen by holding a piece of filter-paper between the tube and a bright flame or window, and looking through the tube at the light. A mere *yellow* colour is often produced in the absence of a nitrate and may be disregarded.

The test is made more delicate by letting the tube stand in a small beaker of cold water for a few minutes, since heat prevents or destroys the brown colour. If much nitric acid is present, on mixing the acid and $FeSO_4$ solution by shaking, the brown colour extends upwards, until by the heat evolved by dilution of the acid the NO is expelled forming brown fumes in the test-tube, and the liquid loses its dark brown colour (244).

248. Place some small scraps of Cu in some KNO_3 solution, and add strong H_2SO_4; brown nitrous fumes appear at once or on warming the tube: they are best seen, if small in quantity, by looking down the tube at a white surface.

The liquid at the same time becomes blue from the presence in it of $Cu(NO_3)_2$:—

$$8HNO_3 + 3Cu = \overline{2NO} + 3CuN_2O_6 + 4H_2O.$$

NO is itself a colourless gas, but yields on mixture with air N_2O_3 and N_2O_4 which are reddish-brown.

249. If some cold dilute indigo-solution be poured into cold KNO_3-solution until it is decidedly blue, and then H_2SO_3 be added, the blue colour remains unchanged (Difference from chlorates).

250. If any solid nitrate be fused for some time at a red heat with fusion mixture on platinum foil, oxygen gas is given off slowly with effervescence* and KNO_2 is formed; if the foil, after cooling, be boiled in water, a portion of the solution may be proved to contain KNO_2 by the test in paragraph (245); another portion, if acidified with HNO_3, will be found to give no precipitate with $AgNO_3$ (Difference from chlorates).

A solution of a nitrate may also be reduced to nitrite by nascent H. Add to a little KNO_3-solution some dilute H_2SO_4 and starch paste with KI, no colour will be produced, but on dropping in a fragment of Zn a deep blue colour is seen (245).

251. Solid KNO_3 fused on charcoal in the blowpipe flame "deflagrates," that is to say, the surface of the charcoal burns rapidly like gunpowder.

251 *a.* Many metallic nitrates [*e.g.* $Pb(NO_3)_2$] when heated in a glass tube closed at one end, evolve reddish-brown fumes with a characteristic smell (N_2O_4) and oxygen—$PbN_2O_6 = N_2O_4 + O + PbO$. The fumes are recognised by their colour and smell, the O by its inflaming a glowing splinter of wood.

Hydrogen nitrate, or *nitric acid* (HNO_3), has a strong acid reaction, it gives with $FeSO_4$ solution a brown ring (247), and with Cu brown fumes (248) without addition of H_2SO_4. Evaporated with quill-clippings, it stains them bright yellow.

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* If this experiment is performed in a small hard glass tube as described in (254), the oxygen can rarely be detected, since it is evolved very slowly: hence the student may perform the ignition on platinnm foil and *neglect* the test for oxygen.

CHLORATES ($-'ClO_3$).—Use $KClO_3$.

252. If to a *cold* solution of $KClO_3$ some cold dilute indigo solution be added drop by drop till the liquid is coloured faintly but distinctly blue, then some H_2SO_3 or $NaHSO_3$ be poured in and the mixture shaken, the blue colour of the indigo is at once destroyed (Difference from nitrates).

253. *Strong* H_2SO_4 poured upon a little solid $KClO_3$ becomes orange-red in colour, and evolves when shaken a bright yellow gas (Cl_2O_4):—

$$3KClO_3 + H_2SO_4 = \overline{Cl_2O_4} + KClO_4 + K_2SO_4 + H_2O .$$

This gas has a smell somewhat resembling that of chlorine; it explodes readily when gently heated, hence on warming the mixture in the tube crackling sounds or small explosions will be produced. This experiment may be dangerous, unless the $KClO_3$ is used in very small quantity, and heated gently with the mouth of the test-tube turned away from the operator.

254. *$AgNO_3$* produces no precipitate with $KClO_3$-solution if it is free from KCl. But if some solid $KClO_3$* be heated in a test-tube, or better in a small ignition-tube (10), after melting it begins to give off oxygen with effervescence; this gas may be recognised by placing in the tube a slip of wood with a spark at its end, which is easily obtained by kindling a match and suddenly blowing out the flame, the spark is caused to burst into a flame. If the tube is then allowed to cool and the residue dissolved by boiling with some water, a portion of the solution will be found to give no reaction for HNO_2 (245); but another portion will give a white precipitate (AgCl) with $AgNO_3$, which does not dissolve in HNO_3 even on boiling, but is easily soluble in AmHO (difference from a nitrate): this precipitate is caused by KCl:—

$$KClO_3 = KCl + O_3 .$$

---

* If the chlorate used for this test does not yield a *soluble* chloride, fusion mixture must be mixed with it before heating, in order to obtain a soluble chloride in the residue.

255. *Blowpipe test.*—$KClO_3$ if heated on charcoal in the blowpipe flame, causes the charcoal to "deflagrate."

Hydrogen chlorate, or *chloric acid* ($HClO_3$), is a colourless, odourless liquid, which first reddens and then bleaches litmus-paper; on being kept for some time it changes into O, Cl, $HClO_4$ and H_2O; the same change occurs rapidly when it is heated.

256. As will be seen, there is little difficulty in distinguishing a nitrate from a chlorate. When present together they more or less interfere with one-another's reactions; but they may be detected, in the absence of chlorides and nitrites, by heating strongly for a short time and testing the residue for nitrite and chloride (250 and 254).

GROUP IV.—CHLORIDE GROUP.

Chlorides, bromides, and iodides closely resemble one another; they are all precipitated by $AgNO_3$ from solutions to which HNO_3 has been added in excess; in this respect they differ from all salts except cyanides, ferrocyanides, and ferricyanides, and these are easily distinguished by other means.

CHLORIDES (–'Cl).—Use NaCl.

257. *$AgNO_3$* added to a little NaCl dissolved in water, gives a *pure white* precipitate (AgCl), which collects into curdy masses when heated or shaken, and quickly turns violet in sunlight or more slowly in daylight. Decant the water; to one portion of the precipitate add HNO_3 and warm, it does not dissolve; to another portion add AmHO, it readily dissolves. Decant the liquid from a portion of the precipitate after shaking and letting the precipitate settle, and warm the precipitate with a little strong H_2SO_4, the acid remains colourless and no coloured vapour is given off.

258. Solid NaCl warmed with strong H_2SO_4 gives off colourless HCl-gas, which is recognised by fuming in the air, reddening moistened blue litmus-paper, and making a drop of $AgNO_3$ solution on the end of a glass rod milky.

259. If solid NaCl be powdered and mixed with finely powdered MnO_2, then strong H_2SO_4 added and the mixture warmed, chlorine gas comes off:—

$$2NaCl + MnO_2 + 3H_2SO_4 = 2\overline{Cl} + MnSO_4 + 2NaHSO_4 + 2H_2O.$$

This gas is recognised by its property of bleaching a piece of moist blue litmus-paper, introduced into the tube on a glass rod. The most delicate way of testing is to warm the mixture in a small beaker covered with a watch glass which bears on its under surface a piece of moist litmus paper, and let the apparatus stand for some time. Moist starch is not coloured when held in the air in the tube.

Many samples of commercial MnO_2 evolve Cl when heated with H_2SO_4; hence the MnO_2, unless specially prepared by precipitation, must be carefully tested before using it for this reaction; or the MnO_2 and H_2SO_4 may be first boiled together as long as any bleaching action is produced when moist litmus is held in the tube, then the substance to be tested is added and Cl again tested for whilst heating the mixture.

260. If an intimate mixture of solid NaCl with three or four times as much $K_2Cr_2O_7$ in fine powder be made by rubbing the two substances together in a mortar, and this mixture be then poured into the tubulated flask shown in fig. 38*, page 132, and mixed with strong H_2SO_4 by pouring in the acid and shaking it round in the flask, on warming the flask reddish-brown vapour (CrO_2Cl_2) will be evolved:—

$$4NaCl + K_2Cr_2O_7 + 6H_2SO_4 = 2CrO_2Cl_2 + 4NaHSO_4 + 2KHSO_4 + 3H_2O.$$

If the neck of the flask be closed by an india-rubber or a glass stopper and the delivery-tube be made to dip into water in a test-tube, the vapour on passing into the water will impart to it a reddish-yellow colour owing to the formation of H_2CrO_4:—

$$CrO_2Cl_2 + 2H_2O = H_2CrO_4 + 2HCl.$$

---

* The test-tube fitted as shown in fig. 37 is much less suitable.

On adding to this yellow liquid excess of AmHO, the colour becomes pale yellow; on now adding excess of $H\bar{A}$, the original reddish-yellow colour is reproduced, and in this liquid the presence of H_2CrO_4, and therefore indirectly that of HCl, may be shown by the formation of a yellow precipitate on addition of $Pb\bar{A}_2$-solution.

Since by this method the detection of a chloride depends on the formation of H_2CrO_4 in the liquid in the test-tube from the vapour of CrO_2Cl_2, great care must be taken that no H_2CrO_4 is introduced into that liquid from any other source. Now, since the mixture in the flask contains a chromate, the greatest precaution must be taken that none of it is allowed to get into the bent delivery-tube, and thence into the test-tube. In transferring the powder to the flask, it must be poured down the side of the *perfectly dry* neck opposite to that at which the delivery-tube enters; also whilst heating the mixture it must not be allowed to rise or splash into the neck of the flask. If these precautions are not taken, and H_2CrO_4 as such is introduced into the water in the test-tube, the test is worthless as an indication of the presence of a chloride.

Care must also be taken that the liquid is not sucked back from the test-tube into the flask: this is prevented by raising the delivery-tube out of the water in the test-tube as soon as the heating is stopped.

Hydrogen chloride or *hydrochloric acid* (HCl) is a colourless gas which fumes in the air, and dissolves very readily in water; both the gas and its solution render $AgNO_3$ solution, which has been acidified with HNO_3, milky. Heated with MnO_2 the liquid acid evolves chlorine gas.

BROMIDES (–'Br).—Use KBr.

261. *$AgNO_3$* added to a little KBr solution gives a *yellowish-white* precipitate (AgBr), which is easily coagulated by heating or shaking the liquid; it is insoluble in HNO_3, but rather soluble in AmHO, thus somewhat resembling the AgCl precipitate, from which it is distinguished by its colour. By shaking well, allowing to settle, decanting the liquid, and heating the precipitate with strong H_2SO_4, no violet vapour is evolved, the precipitate thus differing from AgI.

262. Solid KBr when heated with strong H_2SO_4 gives off *reddish-brown* vapour of Br*, which somewhat resembles Cl in smell, but differs by its colour and by turning moist starch *orange-red*, the colour disappearing by heat. This last test is best performed by dipping the wetted end of a glass rod into some powdered starch, and then moistening the adhering starch by breathing upon it several times. If MnO_2 be mixed with the KBr powder before adding H_2SO_4, the Br is evolved in greater quantity and more readily.

263. *Cl-water*, added carefully drop by drop to a little KBr solution whilst shaking it, liberates Br, which gives the solution an orange-red colour; excess of Cl-water must be carefully avoided, as it destroys the colour :—

$$KBr + Cl = Br + KCl.$$

On warming a part of this solution in a dish, brown fumes are given off which colour *orange-red* some starch held in the vapour as described in the preceding reaction.

If CS_2 be added to another portion and the liquids are then well shaken together, the Br is dissolved by the CS_2, and on standing the reddish brown CS_2 solution sinks to the bottom; on adding a little KHO solution and shaking well, the colour of the Br again disappears from the CS_2, owing to the formation of the colourless salts KBr and $KBrO_3$:—

$$6Br + 6KHO = 5KBr + KBrO_3 + 3H_2O.$$

264. A mixture of solid KBr and $K_2Cr_2O_7$ heated with strong H_2SO_4 as in (260), gives off reddish-brown vapour of Br:—

$$6KBr + K_2Cr_2O_7 + 11H_2SO_4 = 3Br_2 + Cr_2(SO_4)_3 + 8KHSO_4 + 7H_2O.$$

This vapour, if received in a little water, imparts its colour to the water; if the liquid is shaken up with CS_2 however, the Br is dissolved away from the water by the CS_2, which forms a brown layer at the bottom of the water; by shaking well, after addition of AmHO or KHO, the colour of the solution is destroyed (Difference from a chloride).

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* KBr *solution* does not so easily give off Br vapour, but the liquid becomes coloured reddish-brown.

Hydrogen bromide, or *hydrobromic acid* (HBr), is a gas resembling HCl; its solution differs by evolving Br when heated with MnO_2.

IODIDES (– 'I).—Use KI.

265. *$AgNO_3$* added to a little KI solution, gives a *yellow* precipitate (AgI), which is easily coagulated by heating or shaking the liquid: it is insoluble in HNO_3, and very slightly soluble in AmHO.

This precipitate differs from AgCl and AgBr by its colour and its slight solubility in AmHO, but more particularly by its behaviour when heated with strong H_2SO_4. If the precipitate, coagulated by heating or shaking the liquid, is allowed to settle and the liquid decanted, then strong H_2SO_4 poured in and heated with the precipitate, the acid becomes coloured red, and violet vapours of iodine are seen especially on looking down the tube; the colour is particularly distinct after the tube has been allowed to cool for a short time. This vapour of iodine may be detected when almost invisible by holding in the air of the tube a glass rod previously dipped into freshly-made starch solution, which will become coloured blue.

266. *$CuSO_4$* mixed with H_2SO_3 or $FeSO_4$ solution, which reduces it to Cu_2SO_4, gives a white precipitate (Cu_2I_2); warming, or the addition of a single drop of AmHO, causes this precipitate to form more quickly:—

$$2CuSO_4 + H_2SO_3 + H_2O = Cu_2SO_4 + 2H_2SO_4$$
$$2KI + Cu_2SO_4 = Cu_2I_2 + K_2SO_4.$$

If this precipitate is allowed to settle and the water poured off, or if it be filtered, and the precipitate is then warmed with strong H_2SO_4, the liquid becomes violet and gives off violet fumes of I.

Solutions of chlorides and bromides are not precipitated thus by Cu_2SO_4.

267. Solid KI if warmed with strong H_2SO_4 gives off violet vapour of I[*], which, if in large quantity, cools on the sides of the tube to black solid I: this vapour colours starch solution blue, as may be proved by dipping a rod moistened with freshly-made starch solution[†] into it, or by pouring the heavy fumes out into a white porcelain dish whose inside has been wetted with starch solution, or by dipping into the vapour a slip of filter-paper moistened with starch solution. The I is more readily and plentifully evolved if some MnO_2 be mixed with the KI before adding H_2SO_4.

268. *Cl-water* added drop by drop, carefully avoiding excess which would form colourless ICl_3, sets free I, which dissolves in the water giving a brown liquid; divide this into three parts:—

1. Heat one part; violet vapour of I is given off and is best seen by looking down the mouth of the tube at a white surface. The colour is most distinctly seen if, before heating, some strong H_2SO_4 is added to the liquid. If a glass rod is dipped into starch solution and held in the vapour, the starch becomes blue.

2. To another portion starch solution is added, which gives an intense blue colour often appearing black unless much starch is added and the liquid largely diluted; on warming the solution the colour disappears, but often reappears when the liquid is cooled. To insure the disappearance of the colour on being heated, the solution must be diluted and the starch added in excess.

3. To the third portion add a drop of CS_2 and shake well; the CS_2 dissolves the I, and gradually settles to the bottom having a beautiful violet colour. Add a little KHO solution and shake well, the violet colour of the CS_2 will disappear owing to the formation of the colourless salts KI and KIO_3.

The last two methods of testing for the free I are by far the most delicate known; but Cl-water is by no means the best reagent for

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[*] KI *solution* does not so easily give off purple vapour, but the liquid becomes reddish-brown or red.

[†] The starch solution must always be freshly made (par. 526, remark 35), as after being kept it loses the property of yielding a blue colour with iodine.

setting I free from its compounds, because if added in excess it combines with the I and prevents its detection. The reagents described in (269) are much better suited for liberating I from its compounds.

269. If a little KNO_2 is added to the KI solution, and then any dilute acid ($H\bar{A}$, HCl or H_2SO_4) poured in, HNO_2 is produced and liberates I. A solution of N_2O_4 in dilute H_2SO_4, made by heating $Pb(NO_3)_2$ strongly and passing the red fumes into dilute H_2SO_4 (528. 5), also sets free I from KI. The I thus liberated in solution may be identified by the three methods described in (268), of which the third is the most delicate.

These two reagents for liberating I do not hinder its detection if they are added in excess, and they are also useful because they do not liberate Br from its compounds.

270. *$HgCl_2$* gives with a solution of iodide a scarlet precipitate, with a solution of bromide or chloride it gives no precipitate; $Pb\bar{A}_2$ gives with iodide solution a *bright yellow* precipitate, with bromide or chloride a *white* precipitate.

Hydrogen iodide or *hydriodic acid* (HI) is a gas resembling HCl and HBr; its solution differs by evolving I when heated with MnO_2.

271. *A chloride, bromide,* or *iodide,* is easily detected by the foregoing tests. The reactions given in paragraphs (259) and (260) are perhaps the most characteristic tests for a chloride, those described in (262), (263) and (264) for a bromide, whilst an iodide is detected with the greatest ease and certainty by reaction (269).

The colour of the precipitate produced by $AgNO_3$ in the solution made acid with HNO_3, and its behaviour with AmHO, though less reliable than the above tests, serve to indicate which of these acid-radicles is present, AgCl being *pure white* and very easily soluble in AmHO, AgBr being *pale yellow* and not readily soluble in AmHO, whilst AgI is *primrose-yellow* and almost insoluble in AmHO. See also the distinctive action of hot strong H_2SO_4 on AgI, described in paragraph (265).

272. (s) *The detection of a chloride, bromide, and iodide when occurring together.*

The method of examination varies according to whether an iodide is present or absent; since a chloride cannot be detected in the presence of an iodide, the latter, if present, must be separated before proceeding to examine for the chloride (273). In paragraph (274) is given a process for detecting an iodide and bromide; it is of value only when a chloride has not to be tested for.

273. (s) To a small portion of the solution, made just acid if necessary by addition of dilute H_2SO_4, add a little cold freshly prepared starch solution and then strong HNO_3, or either of the reagents mentioned in (269), drop by drop; a dark blue coloration* shows the *presence of an iodide.* The remainder of the solution is examined for a chloride and bromide by the directions given below under *A* or *B* according as an iodide is present or absent:—

A. An iodide is present.—Add to the solution a reagent consisting of a mixture of $CuSO_4$ solution with half its measure of strong H_2SO_3 solution, and warm gently for a short time; Cu_2I_2 will be precipitated (266). In order to be sure that the iodide is entirely precipitated, a small quantity of the liquid must be filtered and warmed with a little more of the above reagent: if any further precipitate is caused, the filtered portion is returned to the unfiltered liquid, more of the reagent is added to the whole, which is then warmed for a time and again tested as above, to see if the precipitation is complete; these trials are repeated until a small portion, on filtration, gives no further precipitate when warmed with more of the reagent. The whole is then filtered till clear, pure NaHO is added in excess to the filtrate and the liquid boiled. The precipitate thus produced is filtered off, and the filtrate, now free from iodide, is tested for bromide and chloride by the directions given under *B.*

B. No iodide is present.—Make the solution alkaline, if not already so, by addition of pure NaHO, then evaporate it to dryness in a porcelain dish, scrape the residue off the sides of the dish and mix it with three or four times as much powdered $K_2Cr_2O_7$ by rubbing the two substances together in the dish with a pestle (260). Transfer this mixture into the small tubulated flask (fig. 38, p. 132) by pouring it down the perfectly dry neck on the side opposite to that at which the tube enters, and taking the greatest care to let none of the powder enter the delivery-tube. Then pour into the flask strong H_2SO_4 sufficient to cover the powder to the depth of about a quarter of an inch, at once close the neck with an india-rubber or glass stopper, and dip the end of the delivery-tube into water contained in a test-tube. Mix the contents of the flask by cautious shaking, and warm the mixture gently, carefully guarding against allowing any of the mixture to get into the delivery-tube and thence into the water in the test-tube*. As soon as no more reddish-brown vapour is visible in the flask or its delivery-tube, discontinue the heating, and immediately withdraw the test-tube. If no coloured vapour has been evolved and the water in the test-tube is colourless, chloride and bromide are absent. If the water is coloured, pour into it CS_2 sufficient to form a layer at the bottom of the tube about as large as a small nut; close the mouth of the tube with the thumb, and shake vigorously: if the CS_2 on settling shows a brown colour, the *presence of Br from a bromide* is shown.

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* Black, unless the starch solution has been added in sufficient quantity to the dilute solution.

Separate the water from the CS_2 by pouring it through a wetted filter, add to the filtrate AmHO in excess, then $H\bar{A}$ in excess, and $Pb\bar{A}_2$ solution. A yellow precipitate indicates the presence of a chromate, which proves indirectly that a *chloride was present.*

273 *a.* (s) The above method will also serve for the examination for chloride, bromide, and iodide in the $AgNO_3$ precipitate. The precipitate is dried, and fused for about ten minutes in a porcelain crucible with three or four times as much fusion mixture; the cool mass is boiled with H_2O for some time and filtered, the filtrate being examined as directed above (273).

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* See the precautions in the note at the end of par. 260.

274. (s) If only an iodide and a bromide have to be tested for, the following method may be employed. Pour into the solution CS_2 sufficient to form a large drop at the bottom; add Cl-water drop by drop, shaking thoroughly after each addition: an iodide, if present, shows first by its liberated iodine colouring the CS_2 purple, but on continuing the addition of Cl-water this colour is destroyed; a bromide is then detected by a brown coloration of the CS_2, which however is also destroyed if too much Cl-water is added.

GROUP V.—PHOSPHATE GROUP.

Phosphates and arsenates resemble one another closely in many reactions; the points of difference they present from one another and from other salts will, however, if carefully attended to, render their detection a simple matter.

Phosphates ($-'''PO_4$).—Use $Na_2HPO_4.12H_2O$ solution.

275. *$MgSO_4$* solution to which some AmCl has been added and then a little AmHO, gives with Na_2HPO_4 a white crystalline precipitate ($MgAmPO_4$): this precipitate is almost insoluble in AmHO, but easily soluble in acids. If very little Na_2HPO_4 is present, the precipitate appears only on warming, stirring, or shaking well, and letting stand.

276. *Fe_2Cl_6* dropped in after a little $H\bar{A}$ and some $Na\bar{A}$ solution gives, especially when warmed, a yellowish-white precipitate of $Fe'''PO_4$.

277. *$AmHMoO_4$* dissolved in HNO_3 gives a yellow precipitate if a drop of Na_2HPO_4 solution is added to it; the formation of the precipitate is hastened by *gentle* warming, stirring, or shaking, but it often appears only after a time. Pour off a part of the liquid and show that the precipitate is soluble in KHO, NaHO, and AmHO; and insoluble in acids, especially in HNO_3.

This reaction (277), if properly performed, is the most delicate test known for a phosphate, but careful attention must be paid to the following precautions:—The $AmHMoO_4$ solution must be prepared according to the directions given in (526, remark 40). The solution to be tested must not be alkaline to test-paper, it is best made distinctly acid with HNO_3; it is then to be added *in small quantity only* to some of the $AmHMoO_4$ solution in a test-tube, more of the liquid being added if after *gently* warming and stirring or shaking well no yellow precipitate forms after a few minutes. This last precaution must be particularly observed, as an excess of phosphate altogether prevents the formation of the precipitate: show that this is the case by pouring into some Na_2HPO_4 solution, contained in a test-tube and made acid with HNO_3, a few drops of $AmHMoO_4$ solution; it will be found that no precipitate will form even on heating and shaking the liquid, since the phosphate is present in large quantity compared with the $AmHMoO_4$; but on adding a few drops of this liquid to some fresh $AmHMoO_4$ solution the precipitate appears. It must also be remembered that HCl retards or prevents the formation of this precipitate, and therefore an HNO_3 solution should always be used when possible.

278. *$AgNO_3$:* yellow precipitate (Ag_3PO_4): pour off a portion and show that the precipitate is soluble in AmHO and in HNO_3.

278 *a*. A very delicate test for the presence of P in the form of a phosphate or otherwise, consists in strongly heating the dry solid substance with a small piece of Na in a little tube of hard glass closed at one end: on breaking the tube and breathing upon the powdered substance a smell of onions, due to H_3P, is perceived.

A phosphate made into a paste with strong H_2SO_4 and heated strongly at the tip of the inner blowpipe flame, gives a bluish green colour to the flame.

Hydrogen phosphate, or *phosphoric acid* (H_3PO_4), is a colourless crystalline substance; its solution is strongly acid, but does not char paper and organic substances (Difference from H_2SO_4). It yields reactions (275 and 277.)

ARSENATES ($-'''AsO_4$).—Use $Na_2HAsO_4.12H_2O$ solution.

279. The precipitates formed in reactions (275, 276, 277) for phosphates are precisely similar in appearance and general properties to those formed with the same reagents by arsenates. The precipitate, however, which is formed by an arsenate with $AgNO_3$ differs from that given by a phosphate by being *brown* in colour; also $AmHMoO_4$ gives with an arsenate a precipitate *only on boiling*.

280. The following differences serve to detect and separate a phosphate and an arsenate:—

1. The fact that an arsenate yields a precipitate with $AmHMoO_4$ only when the liquid is *boiled*, whereas the corresponding precipitate with a phosphate is produced by *gentle heat.*

2. An arsenate solution, if boiled with strong HCl, gives when H_2S is passed into the hot liquid, first a white precipitate of S, then yellow As_2S_3; a phosphate solution, under the same circumstances, gives no precipitate.

3. In a *perfectly neutral* solution, $AgNO_3$ gives with an arsenate solution a *brown* precipitate, with a phosphate a *canary-yellow* precipitate.

Hence, if occurring separately, a phosphate and arsenate may be distinguished by one or more of these differences.

281. The simplest method of ascertaining which of these two acid-radicles is present is to throw the precipitate yielded by AmCl, AmHO, and $MgSO_4$ upon a filter, wash it with a little cold water, and drop $AgNO_3$ upon it: if the precipitate consisted of $MgAmAsO_4$ it will become *brown*, if of $MgAmPO_4$ *canary-yellow* (280. 3). Or the precipitate produced by AmCl, AmHO, and $MgSO_4$ may be examined for arsenate by dissolving a part of it in dilute HNO_3, adding $AgNO_3$ as long as it causes any white precipitate, and then very dilute AmHO gradually drop by drop; if arsenate is present a *brown* precipitate will form just before the liquid becomes alkaline to test-paper: phosphate gives a *yellow* precipitate. Instead of mixing the dilute AmHO it may be poured upon the top of the acid liquid, when the colour appears at the surface of contact.

282. *If both arsenate and phosphate have to be tested for*, AmCl, AmHO, and $MgSO_4$ are added as long as any further precipitate is caused: the precipitate is filtered off* and dissolved in a little boiling strong HCl, H_2S is passed for several minutes into this hot HCl solution, when the presence of arsenate is shown as above (280. 2); the precipitate, if any has formed, is filtered off and H_2S passed into the boiling filtrate; if any further precipitate is produced, the gas must be passed again until it no longer causes a precipitate; the clear filtrate from this precipitate is then evaporated just to dryness, dissolved in a little dilute HNO_3, and tested for phosphate by adding a few drops of it to $AmHMoO_4$ solution and gently warming. Arsenite, if present, may be detected by H_2S in the filtrate from the Mg precipitate, acidified with HCl: yellow As_2S_3 is precipitated at once.

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* A small portion of the precipitate may be filtered off upon another filter and tested at once for arsenate by dropping upon it $AgNO_3$ solution. If the precipitate turns brown, arsenate is present and must be separated in the other part of the Mg precipitate before testing for phosphate: if the precipitate turns yellow, phosphate alone is present.

UNGROUPED ACID-RADICLES.

The salts of the following acid-radicles present in their reactions no marked resemblances to one another or to other salts, and are usually detected by special tests.

BORATES ($-'BO_2$, or $-'''BO_3$).—Use $Na_2B_4O_7.10H_2O$.

283. If some powdered $Na_2B_4O_7$ be stirred with a little dilute HCl on a watch glass and a strip of turmeric paper be half immersed in the liquid, then dried on a watch glass in the steam-oven or on a water-bath*, the part which was moistened appears *reddish-brown*, and becomes *bluish-black* when moistened with AmHO. This is a most useful test for a borate.

284. If some methylated spirit be poured upon a little solid $Na_2B_4O_7$ in a test-tube or porcelain dish, some strong H_2SO_4 added, and the mixture be heated and set fire to, the spirit burns with a green-edged flame, seen best by blowing the flame out after it has burnt for a time and then relighting it. This test is not quite characteristic, since some chlorides yield a somewhat similar green flame.

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* The paper may also be dried over a small flame, if it is not heated above steam-heat (100° C.)

285. A mixture of CaF_2, $Na_2B_4O_7$*, and $KHSO_4$, if finely powdered, moistened, and heated on a loop of platinum wire in the inner blowpipe flame, colours the outer flame momentarily *green* after being heated for a short time.

This test is not always decisive, since many phosphates and Cu-salts give the same coloration, and it is besides liable to be masked by the flame colorations of other substances.

Hydrogen borate, or *boric acid* (HBO_2), is a white porous mass, when combined with $2H_2O$ it forms a crystalline scaly hydrate; when strongly heated these yield a fusible glass consisting of B_2O_3. The solution of the acid reddens litmus-paper and *colours turmeric reddish-brown, especially on drying the paper.* The acid is soluble both in H_2O and in alcohol; on evaporating these solutions, the acid passes off with the vapour of the liquid. Reactions (283, 284, 285), will serve to detect boric acid.

CHROMATES ($-''CrO_4$).—Use $K_2Cr_2O_7$ (or $K_2CrO_4.CrO_3$) solution.

All chromates are more or less yellow or red in colour. The yellow solution of an alkaline chromate becomes orange-red on addition of an acid, owing to formation of a dichromate.

286. Various substances, which readily combine with oxygen, deoxidise acid $K_2Cr_2O_7$ solution to a solution of a chromic-salt, the colour of the solution at the same time changing from *orange-red* to a *bright green.* To show this, add HCl or H_2SO_4 to some $K_2Cr_2O_7$ solution, divide the orange-red solution into five parts and deoxidise each of these portions by one of the following methods:—

a. Pass H_2S into the hot solution; white S separates.

b. Add some $NaHSO_3$ or H_2SO_3 and warm.

c. Add alcohol and boil.

d. Boil for some time, after adding much strong HCl.

e. Add some Zn to the acidified liquid and warm: the change of colour is in this case slow.

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* Boric acid is better, since the Na flame coloration tends to conceal the green coloration caused by BF_3.

In each case the change in composition is shown by the liquid becoming green: the following equations show the decomposition occurring with H_2S and with HCl, the others may be drawn out by the student:—

$$K_2Cr_2O_7 + 3H_2S + 8HCl = Cr_2Cl_6 + 2KCl + 7H_2O + S_3.$$
$$K_2Cr_2O_7 + 14HCl = Cr_2Cl_6 + 2KCl + 7H_2O + 3Cl_2.$$

This green solution gives all the reactions for Cr (102–104).

Note.—Since chromic acid is thus reduced by boiling with HCl and by H_2S to a chromic salt, a chromate leads to precipitation of Cr_2Ho_6 in Group III. in the ordinary course of analysis.

Hydrogen peroxide (H_2O_2), if added after some ether to an acid solution of a chromate, on well shaking the liquids causes the ether to assume a blue colour; addition of a few drops of strong HNO_3 is useful. The H_2O_2 may be produced by adding BaO_2 to the *cold* acid liquid. This reaction is extremely delicate and characteristic.

287. *$AgNO_3$: purple-red* precipitate (Ag_2CrO_4): soluble in AmHO and in HNO_3.

288. *$Pb\overline{A}_2$: yellow* precipitate ($PbCrO_4$): soluble in KHO, insoluble in $H\overline{A}$.

289. *$BaCl_2$:* yellowish-white precipitate ($BaCrO_4$): insoluble in $H\overline{A}$, soluble in HCl.

Hydrogen chromate or *chromic acid* (H_2CrO_4?). CrO_3 dissolves in water to a reddish-yellow liquid, with a strongly acid reaction. The liquid evolves Cl and becomes green if it is boiled with HCl; when neutralised by an alkali it becomes yellow, the solution yielding the reactions in pars. 287, 288, 289.

SILICATES. $\left\{\begin{array}{l}\text{Use } Na_2SiO_3 \text{ solution for liquid; for solid,} \\ \text{finely-ground sand or solid } Na_2SiO_3.\end{array}\right.$

290. *HCl* added slowly drop by drop to a solution of Na_2SiO_3 whilst stirring or shaking it, will usually, unless the solution is very dilute [see note 2 below], give a gelatinous precipitate of H_4SiO_4; but this precipitate is very apt to remain dissolved, hence its non-appearance is no proof of the absence of H_4SiO_4.

290 *a.* Small quantities of H_4SiO_4, or H_4SiO_4 in dilute solution, may be detected by evaporating the solution, made acid with HCl, to dryness in a porcelain dish, and heating the residue to 100° as long as acid fumes come off: on stirring and warming the dry residue with strong HCl, SiO_2 remains undissolved as a white powder.

Note 1.—During the last part of the evaporation the silica often separates as a gelatinous mass, and unless the heat is lessened and the residue stirred with a glass rod it will spirt: spirting is best avoided by finishing the evaporation, as soon as the liquid becomes thick, upon a water-bath. The undissolved SiO_2, left on treating with HCl, is best seen by pouring out the liquid into a test-tube: it is soluble in hot KHO solution.

Note 2.—The solution of Na_2SiO_3 used for these reactions is made of such a strength that acids do not precipitate H_4SiO_4 from it, but by boiling some of it down to a quarter its volume before adding the acid the precipitate is usually obtained.

291. *AmCl* or Am_2CO_3 causes a gelatinous precipitate of H_4SiO_4, which usually forms only after a time.

Blowpipe tests.

292. A little solid SiO_2 or a silicate, if fused into a bead of Na_2CO_3, causes frothing from CO_2 escaping:—

$$Na_2CO_3 + SiO_2 = CO_2 + Na_2SiO_3.$$

293. Fused in a bead of $NaAmHPO_4$ the SiO_2 is not dissolved, but floats about in transparent particles and on cooling remains as little opaque masses in the bead. The particles of SiO_2 are usually best seen in the bead whilst fused.

Hydrogen silicate, or *silicic acid* (H_4SiO_4), is a white gelatinous substance with no reaction to litmus: it is insoluble in water, and but very slightly soluble in acids, but dissolves as an alkaline silicate in solutions of the alkalis or alkaline carbonates. On ignition it forms a white insoluble powder (SiO_2) which yields reactions (292), and (293).

Fluorides (– 'F).—Use finely powdered CaF_2.

The most useful tests for a fluoride depend upon setting free hydrofluoric acid and allowing this to act upon a glass surface, which it "etches" or corrodes, causing it to appear dim when perfectly dry.

294. *Strong* H_2SO_4, if warmed with a little CaF_2 in a test-tube, liberates HF, which causes the glass to behave towards the liquid as if it were greasy. The HF etches the glass, and on washing away the cooled mixture and perfectly drying the inside of the tube, it will be found to be dimmed and roughened.

This test is often performed by gently warming the substance, upon which several drops of strong H_2SO_4 have been poured, upon a watch glass for some time; on washing off the substance and drying the glass thoroughly, it will be found to be dimmed and roughened where the substance and acid rested; this is best seen, if only very slight, by holding the glass so as to see the reflection from its surface.

295. A less simple, but more delicate, plan consists in placing the CaF_2 in a platinum crucible, or in a small leaden cup made by bending a piece of sheet-lead, and pouring upon it some strong H_2SO_4. The crucible is then covered with a watch glass with its hollow upwards and filled with cold water to keep the glass cool; the crucible is placed in a porcelain dish containing sufficient hot water to reach half way up the sides of the crucible, the water being occasionally heated if necessary by a small flame*; on removing the watch glass after ten minutes or more, washing and then thoroughly drying it, the part of the surface covering the crucible will be found to be dimmed by the action of the HF vapours. The delicacy of this method is much increased by the modification described in (296).

296. The convex surface of a watch glass is covered with a thin film of beeswax or solid paraffin by carefully heating the glass held by crucible tongs at some distance above a flame, then rubbing its convex side with a piece of wax or paraffin; it is thus covered with a melted layer, which may be made uniform by once more heating the glass, allowing any excess of wax to drop off, and keeping the glass moving whilst it cools; the coating is finally hardened by immersion for a short time in cold water. Lines or characters are then traced on the glass towards its centre by gentle pressure with the point of a penknife, the object being simply to remove the wax from these parts and not to scratch the glass. The glass thus prepared is exposed as in (295) to the HF vapour for about twenty minutes or half an hour, the wax being prevented from melting by cold water in the hollow of the glass. The watch glass is then removed and cleansed from wax by holding it with crucible tongs above the flame and quickly wiping off the wax when melted: a clean bright surface will be thus obtained on which the traced lines will usually be visible at once on holding the glass up to the light, but if mere traces of a fluoride were present they will be made visible by breathing upon the cold surface.

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* Or the crucible may be heated *gently* over a very small flame or on a *sand-bath.*

297. The presence of SiO_2 or a silicate prevents the liberation of HF by H_2SO_4, SiF_4 being given off instead: this gas possesses no etching power, and therefore if SiO_2 or a silicate is present, the methods given above do not serve for the detection of a fluoride. SiF_4 however produces white fumes in air, and if passed into dilute AmHO yields a colourless flocculent precipitate of H_4SiO_4 and gives AmF in solution: the formation of the flocculent precipitate of H_4SiO_4 is sufficient proof of the presence of a fluoride, but the AmF itself may be detected in the filtrate from this precipitate by adding $CaCl_2$, filtering off the precipitate of CaF_2, and drying and examining it by (296).

By mixing SiO_2 with any substance to be tested for a fluoride the above method may be employed for its detection; use for the reaction a mixture of CaF_2 and SiO_2. A small piece of marble may be dropped into the tube in which the SiF_4 is evolved, to carry that gas over into the AmHO solution.

The changes which occur are thus expressed:—

$$2CaF_2 + SiO_2 + 2H_2SO_4 = SiF_4 + 2H_2O + 2CaSO_4.$$

$$SiF_4 + 4AmHO = H_4SiO_4 + 4AmF.$$

298. *$CaCl_2$* added to NaF solution* gives a gelatinous almost transparent precipitate (CaF_2), which becomes more visible on heating or adding AmHO: slightly soluble in HCl, almost insoluble in $H\bar{A}$.

Hydrogen fluoride, or *hydrofluoric acid* (HF), is a colourless gas which fumes in the air, etches glass and dissolves easily in water. The solution of HF is acid in reaction and differs from all other acids in dissolving SiO_2 and attacking insoluble silicates.

FLUOSILICATES ($-''SiF_6$).—Use H_2SiF_6.

299. *$BaCl_2$:* white precipitate ($BaSiF_6$), which usually appears only on gently warming the solution, and thus differs from $BaSO_4$: it is somewhat soluble in boiling HCl, but insoluble in alcohol. Put by some of this precipitate for reaction (301).

This precipitate differs from the precipitate of $BaSO_4$, which it somewhat resembles in being rather insoluble in HCl, by being more or less transparent and crystalline: but fluosilicates differ from sulphates also in giving no precipitates with $Sr(NO_3)_2$ or $Pb(NO_3)_2$ and in giving, when heated with excess of AmHO, a flocculent precipitate of H_4SiO_4 and AmF in solution (297); fluosilicates differ also by the following very distinctive reactions.

300. *KCl*, on warming, gives a very transparent gelatinous precipitate (K_2SiF_6), which is visible only after being allowed to settle for a time; it is insoluble in alcohol.

301. By evaporating H_2SiF_6 solution, or heating a fluosilicate, with strong H_2SO_4, HF is given off and may be detected by its etching action on glass (294 *et seq.*).

Evaporate a little H_2SiF_6, or $BaSiF_6$ from (299), after adding strong H_2SO_4, upon a watch glass to dryness; on washing off the residue the glass will be found to be etched.

301 *a*. A solid substance (*e.g.* the precipitate of $BaSiF_6$ or K_2SiF_6) may also be examined by any of the methods given above for HF, the most delicate being (296). This method is particularly useful for proving a precipitate produced by $BaCl_2$ to consist of $BaSiF_6$*.

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* Prepared by boiling in water the mass obtained by fusing for several minutes on platinum-foil some powdered CaF_2 and fusion-mixture, *filtering* the solution and acidifying the filtrate with $H\bar{A}$.

302. The above reactions readily distinguish between sulphates and fluosilicates, both of which give precipitates with $BaCl_2$, which are ininsoluble in boiling HCl. There is only one other class of salts—the selenates—which give with $BaCl_2$ a white precipitate insoluble in HCl, and this precipitate ($BaSeO_4$), if boiled with strong HCl for some time, evolves Cl and gradually dissolves as $BaSeO_3$, thus differing from $BaSO_4$ and $BaSiF_6$; further, this HCl solution, if warmed with H_2SO_3, yields a reddish precipitate of Se.

Hydrogen fluosilicate, or *hydrofluosilicic acid* (H_2SiF_6), is a strongly acid liquid, volatilising entirely as $2HF + SiF_4$ when heated in platinum: by evaporating it in glass, the glass is etched: it is precipitated by KCl and by $BaCl_2$.

REACTIONS OF ORGANIC ACID-RADICLES.

The following acid-radicles are included here, although the first four are frequently, and the other three always, classed amongst organic compounds, because they are of such frequent occurrence, and their detection is usually a matter of so little difficulty, that they may reasonably be appended even to an elementary treatise on inorganic analysis.

CYANIDES (– 'Cy).—Use KCy solution.

HCy smells of bitter almonds, the alkaline cyanides also smell faintly of the same.

303. *$AgNO_3$:* white precipitate (AgCy), most easily obtained by pouring a drop of the KCy solution into the $AgNO_3$ solution: show with three separate portions that this precipitate is soluble in KCy solution added in excess, and in AmHO, but insoluble in dilute HNO_3.

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* The readiest means of producing an etch with this precipitate is to pour it off into a watch glass, decant the water, and warm the precipitate for some time with strong H_2SO_4; on washing the glass and drying it, it will be found to be corroded. When the more delicate process (296) has to be employed, the precipitate should be shaken up with the liquid, poured off into a platinum crucible or leaden cup, the liquid decanted, and the precipitate warmed with strong H_2SO_4 as described in (296).

If some of the precipitate be filtered off and well washed with hot water until free from $AgNO_3$ (shown by HCl causing no milkiness with the last few drops of the washing water), then dried, scraped from the filter into a porcelain crucible, and heated strongly, Ag is left: if this residue on cooling be warmed with a little dilute HNO_3, it dissolves, giving off red fumes; and a drop of HCl added to this solution gives a curdy precipitate of AgCl. This reaction distinguishes AgCy from AgCl, AgBr, and AgI, which like it are precipitated by $AgNO_3$ and are insoluble in HNO_3, but which, when dried and strongly heated, melt without decomposing; hence, after cooling, if HNO_3 be warmed with them it dissolves out no Ag, and HCl added to the HNO_3 gives either no precipitate or a mere milkiness due to imperfect washing of the precipitate.

This method is somewhat tedious and need seldom be used, cyanides being readily detected by the following reactions.

304. If some $FeSO_4$ solution, mixed with several drops of Fe_2Cl_6, is added to KCy solution and the liquid is then made strongly alkaline with KHO and boiled, then cooled and HCl added to acid reaction, "Prussian blue" (Fe'''_4Cfy_3*) remains as a deep blue precipitate; or, if in small quantity, it dissolves giving a bluish-green solution. If no cyanide had been present, the addition of HCl would have given a nearly colourless liquid.

305. If to some KCy solution, contained in a test-tube fitted as shown in fig. 37 (p. 132), dilute H_2SO_4 is added, then a small piece of marble dropped in and the liquid boiled, allowing the steam to pass off into some Am_2S solution, AmCyS is formed. This substance gives with Fe_2Cl_6 a *blood-red* coloration, which may be obtained by pouring the Am_2S solution out into a porcelain dish, boiling it, allowing it to cool, making acid with HCl (see Note 1, p. 161), and adding several drops of Fe_2Cl_6: a blood-red colour, only faintly seen when but little KCy is used, shows that HCy has passed into the Am_2S producing AmCyS, and therefore proves the presence of a cyanide.

A little of this red liquid should be added to some $HgCl_2$ solution, if the red colour is destroyed it was certainly due to the presence of AmCyS.

The marble is used to cause evolution of CO_2 gas, which lessens the risk of the Am_2S being sucked back during the reaction.

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* The symbol Cfy conveniently represents the group ($FeCy_6$); it is used here to avoid brackets.

306. The former test can often be more simply performed by boiling the solution of KCy or $HgCy_2$ with Am_2S, which must be added until the yellow colour remains after boiling for a short time. If a dark-coloured precipitate forms, this must usually be filtered off before the colour of the filtrate can be seen. Then filter if necessary, acidify with HCl in a porcelain dish (see Note 1, below), and add Fe_2Cl_6 in small quantity; a blood-red colour is produced in the liquid, which is not removed by heat or addition of HCl, but may be destroyed by adding a few drops of the liquid to some $HgCl_2$.

Note 1.—On adding HCl to the Am_2S (305, 306), white S separates, unless the yellow liquid has been previously boiled in a porcelain dish until it becomes colourless, filling in distilled water as it evaporates; but this separation of S is advantageous, since it helps to show by its whiteness even a faint reddish tint in the liquid. Methods (304, 305, 306) are exceedingly delicate tests for a cyanide. Methods (305) and (306), if less readily performed than (304), exceed it in delicacy. Method (306) is the more easy of the two to perform, and (305) is only used in cases where, after boiling with excess of Am_2S, the filtrate is dark-coloured, and therefore the red coloration would be invisible.

Note 2.—$HgCy_2$ does not show the reactions for a cyanide by methods (303, 304, and 305); but by boiling with Am_2S, according to method (306), AmCyS is obtained in the filtrate from the black HgS precipitate, and will give the red coloration with Fe_2Cl_6.

Solid $HgCy_2$ heated in a small tube closed at one end also gives off cyanogen gas, which burns with a peach-blossom coloured flame.

Hydrogen cyanide, or *hydrocyanic* (*"Prussic"*) *acid* (HCy), is a colourless, volatile, inflammable liquid, smelling like bitter almonds: it is *highly poisonous*, both when inhaled as vapour and swallowed as liquid: it readily mixes with water. The acid is easily detected by $AgNO_3$ (303), also by (304), or by formation of AmCyS when the liquid is boiled with Am_2S (306).

FERROCYANIDES ($-''''FeCy_6$[*], or $-''''\overline{Cfy}$).—Use $K_4FeCy_6.3H_2O$ (or $K_4\overline{Cfy}.3H_2O$) solution.

307. *Fe_2Cl_6:* dark-blue precipitate, "Prussian blue"

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[*] This part of the formula of a ferrocyanide is conveniently written $\overline{Cfy}$ to avoid using brackets.

($Fe_4\overline{Cfy}_3$): insoluble in HCl, soluble in $H_2C_2O_4$ to a dark blue liquid: changed by KHO into brown Fe_2Ho_6.

The solubility in $H_2\overline{O}$ and insolubility in $\underline{HCl}$ are shown by pouring off portions, heating them with HCl and $H_2\overline{O}$ respectively, filtering, and noting whether the filtrate is blue: a third portion is mixed with KHO, and becomes brown.

308. *$FeSO_4$:* light-blue precipitate ($Fe''K_2\overline{Cfy}$), which quickly becomes darker on standing in the air, or immediately on addition of Cl-water or Br-water, or on warming with HNO_3: it is insoluble in HCl.

309. *$CuSO_4$:* a chocolate-coloured precipitate ($Cu''_2\overline{Cfy}$), insoluble in $H\overline{A}$.

310. *$AgNO_3$:* white precipitate ($Ag_4\overline{Cfy}$), insoluble in HNO_3 *and in AmHO.* If heated with HNO_3 this precipitate changes to orange-red $Ag_6\overline{Cfy}_2$, *soluble in AmHO.*

FERRICYANIDES [$-{}^{vi}(FeCy_6)_2$, or $-{}^{vi}\overline{Cfy}_2$].—Use $K_6(FeCy_6)_2$ (or $K_6\overline{Cfy}_2$) solution.

311. *Fe_2Cl_6:* dark-green or brown coloration; no precipitate is formed, as may be seen by adding much water to the liquid.

312. *$FeSO_4$:* dark-blue precipitate, "Turnbull's blue," ($Fe''_3\overline{Cfy}_2$): insoluble in acids: blue colour destroyed by KHO.

313. *$CuSO_4$:* yellowish precipitate.

314. *$AgNO_3$:* orange-coloured precipitate ($Ag_6\overline{Cfy}_2$), insoluble in HNO_3, *soluble in AmHO.* Frequently a white residue is left on adding AmHO, consisting of $Ag_4\overline{Cfy}$, and showing that $K_4\overline{Cfy}$ was present in the $K_6\overline{Cfy}_2$ solution.

SULPHOCYANIDES ($-'CyS$).—Use KCyS solution.

315. *Fe_2Cl_6:* blood-red coloration, but no precipitate: the colour is destroyed by $HgCl_2$ solution, but not by HCl.

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316. Cyanides are usually detected in analysis by methods (304, 305, 306). Ferro- and ferri-cyanides can be found and distinguished by their first three tests, or by the precipitate given by $AgNO_3$ in the solution acidified with HNO_3; this precipitate, if a ferricyanide is present, will have a more or less decided orange-red colour; on filtering, or decanting, and warming the precipitate with AmHO, white $Ag_4\overline{Cfy}$

will be left if a ferrocyanide was present, and on acidifying the filtrate with HNO_3 orange-red $Ag_6\overline{Cfy}_2$ will be precipitated. A sulphocyanide is found by (315).

The three following acid-radicles and their salts differ from the foregoing by being charred and emitting a smell of burning when heated in the solid state; oxalates, however, do so only to a slight extent. If the salt be one of the alkalis or of the alkaline earths, the residue left after ignition contains a carbonate which effervesces with an acid, whereas the original substance did not do so.

OXALATES $(-''C_2O_4,$ or $-''\overline{O})$.—Use $Am_2C_2O_4.2H_2O$, (or $Am_2\overline{O}.2H_2O$).

317. *Strong* H_2SO_4 heated with solid $Am_2\overline{O}.2H_2O$ causes an effervescence of gas consisting of a mixture of CO and CO_2:

$$Am_2C_2O_4 + 2H_2SO_4 = CO + CO_2 + Am_2SO_4 + H_2SO_4.H_2O.$$

The CO is detected by burning with a blue flame when a light is applied, the CO_2 by rendering a drop of lime-water on a glass rod milky.

No blackening or charring is produced by strong H_2SO_4; oxalates differ in this respect from the salts of all other organic acids.

318. *$CaCl_2$, $CaSO_4$, or lime-water*, added to $Am_2C_2O_4$ solution (made by diluting some of the reagent solution in a test-tube with an equal quantity of water), gives a white precipitate (CaC_2O_4), insoluble in AmHO and in $H\overline{A}$, soluble in HCl or HNO_3.

318 *a.* If this precipitate is filtered off, a portion placed on a watch glass will not effervesce with dilute HCl; but if the remainder is dried and heated to dull redness on platinum foil, it is converted without blackening into $CaCO_3$, which effervesces with dilute HCl.

This is a general test for oxalate of K, Na, Ba, Sr, or Ca; oxalates of these metals leave *carbonates* on ignition which effervesce with an acid.

319. Heated with MnO_2 and H_2SO_4, solid oxalates give off CO_2:

$$Am_2C_2O_4 + MnO_2 + 2H_2SO_4 = 2CO_2 + MnSO_4 + Am_2SO_4 + 2H_2O.$$

Since commercial MnO_2 usually contains some carbonate, it will give off CO_2 when treated with H_2SO_4. If the MnO_2 to be used is not known to be free from carbonate the best way to make the test is to add to some of the MnO_2 in a test-tube a little H_2SO_4, and heat to boiling; if CO_2 is found in the tube by a glass rod with a drop of lime-water on its end, continue to boil, occasionally drawing fresh air into the test-tube by pushing down a glass tube near to the surface of the liquid and sucking out the air; as soon as no CO_2 is detectable in the tube after boiling for a short time, and the liquid is distinctly acid, the $Am_2C_2O_4$ is added and the mixture again heated, CO_2 will be rapidly given off, and may be detected by the ordinary methods.

If the MnO_2 gives no CO_2 with H_2SO_4, proceed at once to add the $Am_2C_2O_4$, heat and test for CO_2.

Hydrogen oxalate, or *oxalic acid* ($H_2C_2O_4$, or $H_2C_2O_4.2H_2O$ if crystallised), when heated in a glass tube closed at one end partly sublimes unaltered, and is partly decomposed, yielding vapours which cause coughing; it yields reactions (317) and (319). It dissolves in water to an acid, poisonous liquid.

TARTRATES
($-{}''C_4H_4O_6$, or $-{}''\overline{T}$).

For a neutral tartrate use $Na_2\overline{T}.2H_2O$ or $KNa\overline{T}$; for an acid tartrate use $H_2\overline{T}$ or $NaH\overline{T}$.

320. A solid tartrate or solid $H_2\overline{T}$, when heated strongly in a tube, chars, giving off a smell of burnt sugar.

321. A solid tartrate or solid $H_2\overline{T}$ heated with strong H_2SO_4 blackens, giving off a mixture of CO, CO_2 and SO_2.

322. *$AgNO_3$* gives with solution of a neutral tartrate a white curdy precipitate ($Ag_2\overline{T}$), soluble in $AmHO$ and in HNO_3. Allow some of the above precipitate ($Ag_2\overline{T}$) to settle in a test-tube, and pour off as much of the liquid from above it as possible; then pour very dilute $AmHO$ drop by drop into the tube, until the precipitate, after being shaken up with the liquid, is nearly but *not quite* dissolved: then heat the test-tube gradually and gently by immersing it in a beaker of cold water and slowly raising its temperature to 60°, a brilliant mirror of Ag will form on the interior of the tube. If the liquid, instead of being slowly heated to 60°, be quickly boiled, the Ag deposits as a black precipitate which is not so characteristic.

323. *$CaCl_2$* precipitates from a neutral tartrate solution white or crystalline $Ca\overline{T}$: the precipitate often separates only after shaking the liquid well and letting it stand for a time; it is soluble in acids and its formation is retarded by AmCl and other Am-salts. This precipitate is soluble in strong cold KHO*, is reprecipitated by boiling, but dissolves again more or less completely on cooling.

Decant the liquid from the precipitate and pour upon the precipitate a little very dilute AmHO in a test-tube, drop in a small crystal of $AgNO_3$ and warm carefully as in (322). A silver mirror forms on the part of the glass on which the $AgNO_3$ rests.

324. *KCl, or better $K\overline{A}$,* if added to solution of $H_2\overline{T}$ or an acid tartrate, gives on stirring or shaking a white crystalline precipitate ($KH\overline{T}$). If $H\overline{A}$ is added at the same time, the precipitate may also be obtained with neutral tartrates. Addition of alcohol assists the formation of the precipitate which is soluble in alkalis and in mineral acids.

325. If a few drops of Fe_2Cl_6 solution be added to $H_2\overline{T}$ or $KNa\overline{T}$ solution, then KHO or AmHO until the liquid is strongly alkaline, no precipitate forms. The precipitation of several other metals (*e.g.*, Al, Mn, Cu) by alkalis is similarly prevented by $H_2\overline{T}$: this is owing to the formation of a double tartrate of the alkali and the other metal, which salt is not decomposed by an alkali.

Hydrogen tartrate, or *tartaric acid* ($H_2\overline{T}$), occurs in colourless crystals which do not alter in the air; they taste pleasantly acid, and dissolve in water and in alcohol: they are detected by heating them alone or with strong H_2SO_4 (320, 321): if dissolved and neutralised they also yield the reactions in (322, 323).

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* A milkiness remains, however, if the potash solution contains carbonate.

ACETATES ($-{}'C_2H_3O_2$, or $-{}'\bar{A}$).—Use $Na\bar{A}.3H_2O$.

326. Solid $Na\bar{A}$ if strongly heated blackens, giving off a peculiar smell and a vapour (acetone) which burns with a bright flame when kindled.

327. Heated with strong H_2SO_4, vapour of $H\bar{A}$ is given off, recognised by its pungent smell of vinegar. If alcohol is first added to the $Na\bar{A}$ then strong H_2SO_4, and the mixture is warmed, a fragrant smell of ethyl-acetate ($Et\bar{A}$) is emitted; it is most decided when the liquid is shaken after cooling for a short time.

328. Fe_2Cl_6 added in small quantity to a little $Na\bar{A}$ dissolved in water, gives a deep red colour: on boiling the solution, made neutral but not alkaline with AmHO (if necessary), all the Fe separates as a light-brown precipitate and the liquid becomes colourless. The red colour of the solution before being boiled is changed to yellow by addition of HCl (Difference from the coloration due to sulphocyanide).

Hydrogen acetate, or *acetic acid* ($H\bar{A}$).—"Glacial acetic acid" is at 17° C. a colourless crystalline substance, emitting when heated a very pungent and inflammable vapour: it is strongly acid. It dissolves in water readily, imparting its peculiar smell to the liquid in a mild degree. The acid is completely volatile without decomposition.

329. The above organic acid-radicles may be specially tested for by any of their reactions; (318) being generally used to test for an oxalate in solution (317) or (319) when it is solid; a tartrate is best detected in solution by reaction (322) or (323), or if solid by (320); an acetate is found by (326) or (327).

⁂ After completing the reactions for the acid-radicles the student may proceed to Section VI., unless he is intending to learn the analysis of simple salts, when he proceeds to Section V.

SECTION V.

ANALYSIS OF SIMPLE SUBSTANCES CONTAINING ONE METAL AND ONE ACID-RADICLE*.

Note.—This section may be neglected by a student who is proceeding through the senior analytical course.

330. The ordinary method of proceeding to detect the metal or acid-radicle or both in a simple substance, is to make a few preliminary experiments, which will usually give some idea as to what substance is present, and will occasionally even detect metal or acid-radicle, or both of them, with certainty. Whether the composition of the substance has been satisfactorily proved by this preliminary examination or not, the next process is to endeavour to dissolve the substance, if it is not already in the liquid form, in water or acids: then by adding to the solution certain group-reagents in the proper order of succession, it is found to which Analytical Group the metal present belongs. The following Table gives the analytical groups for metals, each group being headed by its distinctive number, name, and group-reagent:—

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* Two metals may occasionally be present, as when an alum, or tartar-emetic [$K(SbO)\overline{T}$], is given for analysis; also there may be two acid-radicles in the liquids given for analysis, since a salt insoluble in water in occasionally dissolved in an acid: but the student should always be informed of this if it in any way complicates the analysis. It will not usually do so, since in the most commonly occurring case, that of an alkaline earth phosphate dissolved in HCl, the PO_4 is detected in Group III. and accordingly no other acid-radicle is looked for.

Table showing the Analytical

Note.—It will be observed that there are two divisions of Groups II. and III. In analysis it is usual to separate the two divisions of Group II. from one another, after having separated the whole group from the remaining groups; the two divisions of Group III. are by some analysts precipitated together and subsequently separated; they are, however, often precipitated separately, and treated as distinct groups.	Metals whose chlorides are insoluble in water and dilute acids, and which are therefore precipitated from their solutions by hydrochloric acid.	Metals whose sulphides are insoluble in dilute acids, and which are therefore precipitated from *acid* solutions by sulphuretted hydrogen.	
	GROUP I. *Hydrochloric Acid*, or *Silver* Group.	GROUP II. *Sulphuretted Hydrogen* Group.	
		GROUP II.A. *Copper* Group. Sulphides insoluble in NaHO and in Am_2S.	GROUP II.B. *Arsenic* Group. Sulphides soluble in NaHO and in Am_2S.
	[Lead . . . Pb.] Silver . . . Ag Mercurosum . Hg'	Mercuricum Hg'' [Lead . . Pb] Bismuth . Bi Copper . Cu Cadmium . Cd	Tin . . Sn Arsenic . As Antimony Sb
	1.	2.	3.

After discovering to which of the above analytical groups the metal present belongs, it is identified by the properties or appearance of the group-precipitate, and its presence confirmed, if necessary, by special tests made on the precipitate or on another portion of the "original solution*."

The acid-radicle, if it has not been already detected during the examination for the metal, is tested for in a less systematic manner. A few preliminary experiments are first performed, and if these do not detect the acid-radicle, it is found, partly by a systematic examination, including that described above for the detection of the metal, but more commonly by special tests.

A substance insoluble in water and in acids is examined according to the special directions in (367–369).

Since in these analyses only one metal and one acid-radicle are to be looked for, the examination for the metal is carried no further when one metal has been detected with certainty, and the examination for the acid-radicle is similarly at an end

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* By the "original solution" is meant the solution of the substance, or the substance itself, if a liquid is under analysis, to which no reagent *has been* added.

Classification of the Metals.

ls which are precipitated as hydrates m their solutions containing ammo- m chloride when made alkaline with monia, or as sulphides or hydrates en ammonium sulphide or sulphu- ted hydrogen is subsequently added.		Metals whose carbonates are precipitated by addition of ammonium carbonate in the presence of ammonium chloride.	Metals whose solutions are not precipitated by any of the foregoing group-reagents in the presence of ammonium chloride.
GROUP III. *Ammonia* and *Ammonium-sulphide* Group.		GROUP IV. *Ammonium-carbonate* or *Barium* Group.	GROUP V. *Potassium* Group.
ROUP III.A. *monia* or *Iron* Group.	GROUP III.B. *Ammonium sulphide* or *Zinc* Group.		
inium . Al . . Fe mium . Cr	Zinc . . Zn Manganese . Mn Nickel . . Ni Cobalt . . Co	Barium . . Ba Strontium . Sr Calcium . . Ca	Ammonium . NH_4 Potassium . K Sodium . . Na Magnesium . Mg
4.	5.	6.	7.

when the presence of one such radicle has been satisfactorily proved. It must, however, be understood that substances given for analysis are liable to contain impurities, which will yield only slight indications; if in the course of analysis, therefore, *only a slight* result is obtained, proceed until some substance is detected in larger quantity, entering against the slight result "Trace of ——."

The separation and washing of a precipitate by "decantation" (23 *b*, 24 *b*) is recommended wherever it is possible, as this process is more easily performed than that of filtration (23 *a*): if a precipitate, however, refuses to settle after heating and shaking the liquid, filtration must usually be resorted to.

Caution.—In every analysis each result must be fully entered in the note-book *as soon as the experiment has been performed.* The form of entry being the same as that adopted for the directions in the text.

For examples of substances to be given for analysis by Section V. see par. 537.

⁂ The numbers in brackets refer to the paragraphs in the text, in which tests or processes to be employed by the student are fully described.

DIRECTIONS FOR THE ANALYSIS OF A SIMPLE SUBSTANCE.

The substance given for analysis may be either a solid or a liquid. If it is a solid examine by (331 *et seq.*) omitting (334), if a liquid is to be analysed begin at (334).

THE SUBSTANCE GIVEN FOR ANALYSIS IS A SOLID.

331. Make a careful examination of the properties of the substance, noting down its colour*, its smell, if it possesses any, also its form—whether it is in powder, crystals, or non-crystalline pieces: note carefully also any appearance or property which may strike you on examining the substance with the naked eye or with the assistance of a lens: after noting down the results of this examination, the substance is powdered as finely as possible.

If the substance possesses a metallic lustre or appearance, work as directed under (366): if it is not metallic in appearance, proceed to ascertain its solubility by the directions contained in (332), and, according to the result thus obtained, conduct the further analysis of the substance by (333) or by (367).

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* The salts of the following metals are coloured:—Cu, *blue;* Fe''', *brownish-yellow;* Fe'', *pale-green;* Ni, Cr, $CuCl_2$, *green;* Mn, *delicate-pink;* Co, *reddish-pink.* Chromates, *light-yellow;* dichromates, *orange-red;* Sb_2S_3, CuO, MnO_2, *black;* HgO, Pb_3O_4, *red;* PbO_2, *dark-brown;* PbO, *light-brown;* Cr_2O_3, *green.*

SOLUBILITY OF THE SUBSTANCE.

332. Since the method of examination of a solid substance depends upon whether it is soluble or insoluble in water or acids, the following experiments are tried.

Place a small quantity of the finely powdered substance in a broad test-tube or a boiling-tube, fill the tube about one third with water, and heat to boiling :—

<table>
<tr>
<td rowspan="4">1. <i>The substance dissolves.</i>
After examining a portion of the solid substance by the preliminary tests (333) and (335–338 <i>a</i>) proceed to examine the colour and reaction of the liquid to litmus by (334, I., II.), and, refer to (339) for directions for analysis.</td>
<td colspan="4"><i>The substance does not dissolve:</i> heat a fresh portion with dilute HCl to boiling, if it does not dissolve decant and boil the residue with strong HCl:—</td>
</tr>
<tr>
<td rowspan="3">2. <i>The substance dissolves.</i>
<i>Absence of Group I.</i>, except possibly Pb.
After examining a portion of the solid substance by the preliminary tests (333) and (335–338 <i>a</i>) proceed to examine the HCl solution (after noting its colour, see 334, I.) for the metal by (339).</td>
<td colspan="3"><i>The substance does not dissolve:</i> put aside the test-tube containing the substance with HCl, and stir a fresh portion of the substance with a little dilute HNO_3; if it is not dissolved boil. If dilute HNO_3 does not dissolve it, heat with a little strong HNO_3:—</td>
</tr>
<tr>
<td rowspan="2">3. <i>The substance dissolves.</i>
After examining a portion of the solid substance by the preliminary tests (333) and (335–338 <i>a</i>) examine the HNO_3 solution (after noting its colour, see 334, I.) for the metal by (339) taking care after testing for Group I. by HCl to boil down considerably in a porcelain dish after adding a little strong HCl*, and then add dilute HCl before testing for Group II. by H_2S.</td>
<td colspan="2"><i>The substance does not dissolve:</i> mix together the contents of the two tubes in which the substance was heated with HCl and with HNO_3, and heat the mixture:—</td>
</tr>
<tr>
<td>4. <i>The substance dissolves.</i>
After examining a portion of the solid substance by the preliminary tests (333) and (335–338 <i>a</i>) examine the solution in HNO_3 and HCl mixed as directed in the preceding column (332, 3) for the metal by (339).</td>
<td>5. <i>The substance does not dissolve.</i>
Examine a fresh portion of the substance by (367 <i>et seq.</i>).</td>
</tr>
</table>

* This method of proceeding removes HNO_3, which would cause with H_2S a deposit of S.

PRELIMINARY EXAMINATION OF A SOLUBLE SOLID.

333. The Table is thus arranged: in the first column is placed the experiment to be made; in the second a list of the results, any one or more of which may be observed; and in the third the inferences to be drawn from the results. Confirmatory or supplementary experiments are occasionally entered, they are, however, only to be tried when the result preceding them in the second column has been noticed.

A more complete preliminary examination will be found in (387–408).

Experiment.	Observation.	Inference.
I. Heat a portion of the substance in an ignition-tube, or in a small dry test-tube, first gently and at last to redness.	**I. The substance does not change**	**Absence of volatile, fusible and organic substances, and of water.**
	II. The substance changes:—	
	1. Drops of water condense in the top of the tube.	**Presence of H_2O.**
	2. The substance fuses easily	**Probable presence of a salt of Na or K, or of Ba, Sr, Ca, Mg**
	3. The substance sublimes	**Presence of a salt of NH_4, As, or Hg: possibly also of $H_2C_2O_4$, of S (yellow), and of I (purple vapour).**
***Confirmatory.* — Boil another portion of the substance with KHO solution.**	**A smell of NH_3 is evolved and white fumes appear when a rod moistened with strong HCl is held to the mouth of the tube.**	**Presence of NH_4. Confirm by $PtCl_4$ (52), and proceed to (335).**
***Confirmatory.* — Mix another portion of the substance with about three times as much powdered Na_2CO_3 and KCy and heat in an ignition tube.**	**A grey sublimate of minute metallic globules, best seen by a lens.**	**Presence of Hg. Pour upon the original substance a few drops of AmHO, if it blackens Hg′ is present, if not Hg″ is present.**
	A black mirror-like sublimate	**Presence of As.**
	4. The substance blackens, and gives off CO_2, which makes a drop of lime-water on a glass rod milky.	**Presence of an organic acid-radicle, probably $\bar{A}$, $\bar{O}$ or $\bar{T}$: the last is known by a smell of burnt sugar.**
	If the cool residue in the tube effervesces with HCl, whereas the original substance did not.	**The organic acid-radicle is united with K, Na, Ba, Sr, Ca, or Mg.**

Experiment.	Observation.	Inference.
	5. Gas or vapour is given off: smell the vapour:—	
	Red nitrous fumes, recognized by their smell.	From nitrate of a heavy metal.
	SO_2, known by its smell and by turning $K_2Cr_2O_7$ solution green.	From combustion of free S or a sulphide; possibly also from a sulphate, sulphite, or theiosulphate.
	Cl, Br, I, known by smell and colour: Br and I by action on starch paste, and Cl by bleaching moist litmus.	From certain chlorides, bromides, or iodides, also free I.
	A slip of wood with a spark at its end, glows brightly or bursts into flame.	O from chlorate or nitrate, or N_2O from decomposition of NH_4NO_3.
	The gas can be lighted and burns at the mouth of the tube; note the colour of the flame:—	
	Pale greenish-yellow; the gas burns explosively, and nitrous fumes are seen.	Presence of NH_3, from strongly heated NH_4NO_3.
	Bright white, the unburnt gas smells of garlic.	Presence of PH_3, probably from a hypophosphite.
	Peach-blossom coloured flame, grey sublimate of Hg.	Presence of Cy, from $HgCy_2$.
	6. The substance changes colour:—	
	Yellow, hot. *White*, cold.	ZnO
	Yellow-brown, " *Yellow*, "	PbO, SnO_2 or Bi_2O_3
	Dark red or *brown*, " *Red brown*, "	Fe_2O_3
II. Dip into the powdered substance a moistened loop of platinum wire, and hold the loop in the Bunsen flame; moisten with strong HCl, and again hold in the flame.	1. The flame is not coloured . . .	Probable absence of the substances enumerated below.
	2. The flame is coloured:—	
	Intense yellow *	Presence of Na.
	Pale-violet *, appearing *crimson-red* through the indigo-prism . .	" K.
	Red Appearing *green* through the indigo-prism . . .	" Ca.
	Appearing *intense-red* through the indigo-prism . .	" Sr.
	Green *Yellowish-green*	" Ba.
	Bright-green, with *blue* centre after moistening with HCl.	" Cu.
	Green	" B_2O_3.
	Blue *Pale-blue (livid)*	Presence of As, Sb, Pb.
	Vivid-blue	" $CuCl_2$.

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* If the coloration for Na or K is intense, no further examination for the metal is necessary but the presence of K should be confirmed by stirring with $PtCl_4$ (47). Proceed to (335). If the coloration is slight, a trace only of the metal is present, and the examination for another metal is continued.

Experiment.	Observation.		Inference.
III. Heat a portion of the substance in a cavity scooped on charcoal, in the blowpipe flame.	1. The charcoal "deflagrates," or burns rapidly.		Presence of a nitrate or chlorate.
	2 A white mass is left in the charcoal which does not fuse, and shines brightly when strongly heated.		Presence of Ba, Sr, Ca, Mg, Al, or Zn.
Confirmatory. — Detach a small portion of the white mass, place it on red litmus paper and moisten it with a drop of water.	The red paper becomes blue in a short time where the substance rests.		Presence of Ba, Sr, Ca, or possibly of Mg.
Confirmatory.—If the moist residue is not alkaline to test-paper, moisten the remainder on the charcoal with $Co(NO_3)_2$ solution and heat again strongly in the blowpipe flame.	A *blue* residue		Presence of Al, or possibly of a phosphate, silicate, or borate.
	A *pink* residue		Presence of Mg.
	A *green* residue		,, Zn.
	3. The residue is not white and does not consist of globules of metal, neither is there any incrustation formed upon the charcoal; proceed to Exp. IV.		
	4. The residue contains metallic globules or an incrustation forms on the charcoal; proceed to Exp. V., omitting Exp. IV.		
IV. Fuse a small quantity* of the substance into a clear colourless borax bead first in the inner then in the outer flame, noting in both cases the colour of the bead whilst hot and when cold.	The bead is colourless when heated in both flames.		Absence of the metals below.
	The bead is coloured :—		
	In outer flame.	In inner flame.	
	Blue, hot; *green*, cold.	*Red* or *colourless*	Presence of Cu.
	Blue, hot and cold.	*Blue*, hot and cold	,, Co.
	Brown or *dingy-purple*, hot; *light-brown*, cold.	*Grey* or *black*, hot and cold . .	,, Ni.
	Brown, hot; *yellow*, cold.	*Bottle-green*, hot and cold . .	,, Fe†.
	Green†, hot and cold.	*Green*, hot and cold	,, Cr.
	Purple, hot and cold.	*Colourless*, hot and cold . .	,, Mn.
Confirmatory. — The presence of Cr or Mn may be confirmed by fusing a portion of the substance on platinum foil or wire with Na_2CO_3 and KNO_3.	A *yellow* mass on cooling		,, Cr.
	A *bluish-green* mass on cooling . . .		,, Mn.

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* It is necessary to adjust carefully the quantity of substance fused into the bead; if too much is used, the bead will often appear opaque; if too little is employed, one of the metals may escape detection. It is best at first to fuse only a minute quantity into the bead, adding more if no colour, or but a faint colour, is produced.

† Cr if present as a chromate gives a bead *brown* whilst hot, after having been fused in the outer blowpipe flame; but this brown colour is not reproduced in the case of Cr when the bead, after becoming green by being heated in the inner flame, is again heated in the outer flame.

Experiment.	Observation.		Inference.
V. Mix the substance in a small cavity on wood-charcoal with Na_2CO_3 and KCy, and heat strongly in the inner blowpipe flame for several minutes. If metallic globules are obtained, detach one and strike it sharply on the bottom of an inverted mortar with the pestle, and note whether it is crushed to powder (*brittle*) or merely flattened out into a cake (*malleable*). If the globules are white and malleable, take one upon the point of a penknife and see if it will mark paper as a black-lead pencil does.	Metallic scales or globules are obtained, or an incrustation on the charcoal:—		
	Globules.	*Incrustation.*	
	White and *brittle*.	*White* . . .	Presence of Sb.
	White and *brittle*.	*Yellow* . . .	,, Bi.
	Red and *malleable*.	*None* . . .	,, Cu.
	White and *malleable* marking paper easily. . .	*Yellow* . . .	,, Pb.
	White and *malleable* not marking paper, and readily fusible.	*None* . . .	,, Sn.
	White and *malleable* not marking paper, fusible only with difficulty.	*None* . . .	,, Ag.
	None. . . .	*Yellow* whilst hot, *white* when cold; becomes green when moistened with $Co(NO_3)_2$ solution and reheated in the outer flame.	,, Zn.
	None. . . .	*Brown* . . .	,, Cd.
	None. . . .	*White;* on smelling the charcoal a smell of onions is perceived.	,, As.

After the completion of the preliminary tests for the metal, a solution of the substance is made according to the directions in (332), and this solution is examined as there directed.

334. PRELIMINARY EXAMINATION OF A LIQUID.

Experiment.	Observation.		Inference.
I. Note the colour of the liquid. *Note.*—If the colour has to be observed by night it should be examined by the white light obtained by burning a piece of magnesium-ribbon. Delicate tints are best seen by looking through some thickness of the liquid at a sheet of perfectly white paper.	*Pink*	Very delicate	Presence of Mn or dilute Co solution.
		Intense	Presence of Co.
	Green	Very delicate	„ Fe'', or Ni or Cr in dilute solution.
		Intense	„ Ni, Cr, or Cu.
	Yellow	Reddish-yellow	„ Fe''' or a bichromate.
		Light yellow	„ neutral chromate.
	Blue		„ Cu.
	Violet or *purple*		„ Cr or a permanganate.
	The liquid is colourless		Absence of Co, Ni, Cr, Cu, &c.
II. Dip pieces of red and blue litmus-paper into the liquid.	*It is neutral* (Not affecting the colour of either paper.)		Absence of free acids and alkalis, and of all salts but some of those of Am, Na, K, Mg, Ba, Sr, Ca, Ag.
	It is acid (Turning blue paper red. If, on adding Na_2CO_3 to a part of the solution and warming, no effervescence occurs, free acid and acid salt are absent.		Presence of a free acid, of an acid salt, or of a salt with acid reaction.
	It is alkaline (Turning red paper blue.) If the colour of the paper becomes only slightly changed it indicates, probably, the presence of an alkaline salt.		Presence of a hydrate of K, Na, Am, Ca, Sr, or Ba, or of a salt with alkaline reaction.

Experiment.	Observation.	Inference.
III. Evaporate (21) some of the liquid upon a thin watch glass* heated very gently by supporting it some inches above a small flame upon a piece of wire gauze, or by means of a sand or water bath. If a residue remains on the glass bring this gradually into the flame and heat it more strongly.	*A residue is left.* . . . . . On being strongly heated the residue blackens and emits a smell of burning . . The smell resembles that of burning sugar. *Note.*—If the cold residue after ignition effervesces with a drop of HCl, whereas the unignited residue did not, the organic acid-radicle is united with Ba, Sr, Ca, Mg, Na, or K. The results in Exp. I., par. 333, may be here observed and should be noted. Keep this residue for Exp. VI. *No residue is left* . . . . . If the liquid is also colourless, tasteless, and without action on litmus-paper, it must consist only of distilled water, and no further examination of it need be made. If the liquid is alkaline, NH_3 must be looked for; if it is acid, H_2CO_3, H_2SO_3, HCl, HNO_3, H_2SO_4, and other volatile acids only need be tested for.	Presence of some dissolved solid. Pres. of $\overline{A}$, $\overline{O}$, or $\overline{T}$. Pres. of $\overline{T}$. Absence of any dissolved solid.
IV. Add excess of strong NaHO or KHO solution and boil (51).	A smell of NH_3; moist red litmus is turned blue, and a rod moistened with strong HCl produces white fumes when held in the mouth of the test-tube.	Presence of NH_4. No further test need be tried for a metal. Proceed to (335).
V. Experiment II. (page 173) is then tried by dipping the platinum wire into the liquid, concentrated, if necessary, by evaporation.		
VI. Experiment IV. (page 174) is tried by fusing some of the residue from Exp. III. (above) in the borax bead.		

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* A piece of thin glass from the side of a broken flask is a cheap and useful substitute for a watch glass.

PRELIMINARY EXAMINA

335. Pour dilute H_2SO_4 in excess upon the substance in a test-tube

336. A gas is evolved, shown by effervescence or by a characteristi

1. *Colourless gas without smell.* A drop of lime or baryta-water, held in the tube upon the end of a glass rod becomes milky :— *Presence of a carbonate*	2. *Gas smelling of burning sulphur.* A drop of $K_2Cr_2O_7$ solution held in the tube upon the end of a glass rod, becomes green :— *Presence of a sulphite.*	3. *Gas with a most fetid smell.* A slip of filter-paper moistened with alkaline solution of $Pb\bar{A}_2$, and held in the tube, is blackened :— *Presence of a sulphide.*	4. *Yellow gas sme ing of Cl.* A slip of moi litmus-paper he in the tube bleached :— *Presence of a hyp chlorite.*
	2 *a.* The SO_2 gas is given off only after standing for a time or on warming, and yellow S separates :— *Presence of a theiosulphate.*		

337. Add strong H_2SO_4 in small quantity to a fresh portion of the of the results enumerated below occurs*, if none of them is noticed

1. *Fumes are evolved with pungent smell and acid reaction to litmus-paper, the liquid is not coloured* :— *Presence of a chloride or nitrate.*		2. *The liquid becomes yello or red*, and a *chlorous sme is given off:* on warmin the tube a crackling nois or slight explosion is pro duced :— *Presence of a chlorate.*
Confirmatory.—Hold in the fumes a glass rod moistened with a solution of $AgNO_3$ acidified with HNO_3: the solution on the rod becomes milky :— *Presence of a chloride.* Confirm by (351).	*Confirmatory.*—Drop into the acid some small pieces of Cu, reddish-brown gas is evolved :— *Presence of a nitrate.*	*Confirmatory.*—To a portio of the cold solution of th substance in water ad a few drops of dilut indigo solution, then som H_2SO_3, the blue colour o the indigo is destroyed.

338. After heating the tube containing the strong H_2SO_4 and porcelain dish for (338*a*); rinse out the tube, and dry the inside *fluoride* is shown. The presence of a fluoride, if decisively indicated

338*a*. Add to the liquid in the porcelain dish alcohol, mix by tinge it shows :—*Presence of a borate* (confirm by 353).

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* If Pb, Ba, Sr, or Ca is present the addition of H_2SO_4 causes a

TION FOR THE ACID-RADICLE.

and observe the effect produced*; then heat to boiling :—
smell; see below :—

5. *Reddish-brown fumes are evolved,* best seen by looking down the tube at white paper. On adding a little *cold dilute* H_2SO_4 and $FeSO_4$ solution to a fresh portion, a deep-brown liquid :— *Presence of a nitrite.*	6. *Smell of HCy.* Test for a cyanide and a ferro-, ferri-, and sulpho-cyanide in the original substance by (351).	*No gas is evolved* :— Absence of carbonate sulphite, theiosulphate, sulphide, hypochlorite, nitrite. Pass on to (337).
	7. *Smell of vinegar.* Refer to (337, 4) below	

substance, mix well by shaking, heat the liquid and note whether any
proceed to (338) and (338 *a*) :—

3. *A reddish-brown* or *violet* colour is produced in the acid, and no explosion or crackling occurs on warming :— *Presence of a bromide or iodide.* If on heating the mixture, coloured vapour is evolved (see note), hold in the vapour the end of a glass rod covered with moist starch powder: the starch becomes brown :— *Presence of a bromide.* The starch becomes bluish-black :— *Presence of an iodide.* *Note.*—The vapour is much more easily obtained by mixing the substance with MnO_2 powder, before adding H_2SO_4 and heating.	4. *A smell of vinegar* is noticed :— *Presence of an acetate.* *Confirmatory.* — Add to a fresh portion of the substance some alcohol, then strong H_2SO_4; a fragrant smell proves an acetate.	5. *A mixture of CO and CO_2 gases is given off:* found by a drop of lime-water held in the tube being turned milky, and by the CO burning with a blue flame when kindled :— *Presence of an oxalate.*	6. *The substance blackens* and evolves gases, amongst which SO_2 is recognised by its smell and its action on $K_2Cr_2O_7$ solution :— *Prob. presence of a tartrate.* Confirm by (354).

substance, let it stand for some time, then pour the liquid into a
thoroughly; if it is seen to be corroded and dimmed :—*Presence of a*
here, requires no confirmation.
stirring, heat the dish and kindle the alcohol, if the flame has a greenish

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precipitate of the insoluble sulphate: this may be disregarded.

339. GENERAL TABLE FOR THE

If the metal has not been detected in the Preliminary Examination, (see Table pp. 168, 169), and then trying special tests for each

<table>
<tr><td colspan="3">A part of the solution of the substance, <i>if acid</i> (340), is mixed with dilute HCl (341); if</td></tr>
<tr><td rowspan="3">The precipitate may be —
AgCl—white.
Hg_2Cl_2— ,,
$PbCl_2$— ,,
Examine for the metal by Table I. (344).</td><td colspan="2">Into the same part of the solution (342) H_2S is passed, or H_2S-is heated to boiling :—</td></tr>
<tr><td rowspan="2">The precipitate may be—
HgS—black.
PbS— ,,
Bi_2S_3— ,,
CuS— ,,
CdS—yellow.
SnS—brown.
SnS_2—yellow.
Sb_2S_3—orange.
As_2S_3—yellow.
Examine for the metal by Table II. (345).</td><td>To a <i>fresh portion</i> of the solu-
[<i>Caution.</i>—If a tartrate or evaporating the liquid must dilute HCl, and tested by
If AmCl and AmHO produce and warmed. Should a (343) or fluoride (338) have</td></tr>
<tr><td>The precipitate may be—
Al_2Ho_6—{ Colourless and transparent.
Cr_2Ho_6—pale-green.
Fe_2Ho_6—reddish-brown.
$FeHo_2$—{ dingy - green, turning brown in the air.
Examine for the metal by Table III.A (346).
If the precipitate is opaque or white and has none of the above appearances, refer to (357).</td></tr>
</table>

If no metal can be found, the substance is probably an acid (see 334, II.).

NOTES TO THE GENERAL TABLE.

340. If the liquid is *neutral* or *alkaline*, a portion of it is made just acid by slowly dropping in dilute HNO_3; if this produces no precipitate or a precipitate which dissolves on adding more HNO_3, another portion of the liquid is examined by the above Table.

If a precipitate forms which is not dissolved by adding more HNO_3, it may consist of colourless, gelatinous H_4SiO_4 from a silicate, or finely divided S which is precipitated *white* from a polysulphide, and *yellow* from a theiosulphate; or of SnO_2, or metastannic acid, or Sb_2O_5 (all white); it may also possibly be SnS_2, As_2S_3 (both yellow), Sb_2S_3 (orange-red), these sulphides being precipitated from a sulphostannate, sulpharsenite, or sulphantimonite.

If H_4SiO_4 separates it is best to evaporate the acidified solution quite to dryness, then heat the residue with dilute HCl; any insoluble

EXAMINATION FOR THE METAL.

it is most rapidly found by ascertaining to which Group it belongs member of the Group.

<table>
<tr><td colspan="4">any precipitate forms, more HCl is added and the liquid heated :—</td></tr>
<tr><td colspan="4">water is added to it (343), the liquid is then diluted, and if no precipitate has formed it</td></tr>
<tr><td colspan="4">tion AmCl is added, then AmHO in excess :—
oxalate has been found by (337), some of the solid substance or the residue obtained by
be strongly heated on a piece of porcelain, the residue on cooling is then dissolved in
AmCl and excess of AmHO.]
a precipitate, a few drops of the original solution are added to some $AmHMoO_4$
yellow precipitate form, indicating the presence of a *phosphate*, or should a *chromate*
been found refer to (357) for the examination of the Group IIIA. precipitate.</td></tr>
<tr><td colspan="4">To the same portion add a few drops of Am_2S, or H_2S-water :—
[*Note*—Before adding Am_2S the liquid may be *blue*, showing *presence of Ni*, or *violet* from dissolved Cr_2Ho_6; the latter must be precipitated by long boiling in a porcelain dish before adding Am_2S.]</td></tr>
<tr><td rowspan="3">The precipitate may be—
ZnS—white.
MnS—pink.
NiS—black.
CoS— „
[FeS— „
Examine for the metal by Table III.B. (347).</td><td colspan="3">To the same portion add Am_2CO_3 :—</td></tr>
<tr><td rowspan="2">The precipitate may be :—
$BaCO_3$— white.
$SrCO_3$— „
$CaCO_3$— „
Examine for the metal by Table IV. (348).</td><td colspan="2">To the same portion add Na_2HPO_4, warm gently and shake well :—</td></tr>
<tr><td>A white crystalline precipitate proves *presence of Mg*.</td><td>The metal still present may be NH_4, Na, K; it should have been detected already in the Preliminary Examination by Tests I. or II. (333).</td></tr>
</table>

residue confirms the presence of a silicate, it is filtered off, and the filtrate tested for the metal by the above General Table.

Any other substance insoluble in dilute HNO_3 is filtered off and may generally be known by its appearance: further, SnO_2 becomes *yellow* and Sb_2O_5 *orange* when moistened with H_2S water, metastannic acid becomes golden-yellow if moistened with $SnCl_2$ solution.

341. Make a note of any changes which occur on the addition of HCl; gases may be evolved (336), or a *yellow* solution may become *reddish-yellow*, which renders probable the presence of a neutral chromate.

342. Before H_2S is added, the liquid must be heated, and if any smell of Cl or of SO_2 or brown nitrous fumes should be perceived, the solution must be boiled down nearly to dryness, then dilute HCl added and H_2S added at once. During evaporation gelatinous H_4SiO_4 may separate (340).

343. The following changes occurring on the gradual addition of H_2S

are of importance. If the precipitate is *white* at first, then *brown*, and at last *black*, the presence of Hg'' is shown ; if the solution is strongly acid and gives a *red* precipitate becoming *black* on dilution with H_2S-water Pb is present.

A separation of white S may occur accompanied by the following changes of colour, which are characteristic and should be noted down :—

A *reddish-yellow*	solution	changing to	*pale green*	indicates	a ferric salt.
A *reddish-yellow*	„	„	*bright green*	„	chromate.
A *green*	„	„	*colourless*	„	manganate.
A *purple*	„	„	*colourless*	„	permanganate.

The precipitate of S is known by being perfectly white and running through a filter, it need not be further examined.

A yellow precipitate which forms only on boiling the liquid may be As_2S_3 from an arsenate, or SnS_2 from a stannic salt.

344. TABLE I.—SILVER GROUP.

A white precipitate forms on addition of HCl, and is either not dissolved when boiled with HCl, or it dissolves and reappears in crystals on cooling, the liquid showing the *presence of Pb.* The precipitate may consist of AgCl, $PbCl_2$, Hg_2Cl_2.

Let the precipitate settle and decant the liquid : boil the precipitate in the test-tube with AmHO :—

1. *The precipitate dissolves*, at least in part (see note below):— *Presence of Ag.* Confirm by adding excess of HNO_3 to the AmHO solution, a milkiness appears. Note.—Coagulated AgCl often dissolves only slowly in AmHO.	2. The precipitate does not dissolve:—	
	The colour of the precipitate is unchanged:— *Presence of Pb.* Confirm by decanting the liquid, dissolving the precipitate in $H\bar{A}$, and adding K_2CrO_4 to the solution; a yellow precipitate forms.	*The precipitate becomes black:—* *Presence of Hg'* *(Mercurosum).* Confirm by immersing a bright slip of copper in a portion of the original solution, made acid with a few drops of HNO_3; a grey deposit forms on the Cu, which becomes white and bright when rubbed.

345. TABLE II.—COPPER AND ARSENIC GROUPS.

Note the colour of the precipitate yielded by H_2S :—

<table>
<tr><td colspan="4">1. <i>The precipitate is dark-brown or black</i> ; it may consist of SnS, CuS, HgS, Bi_2S_3, PbS .
Add pure NaHO in excess to a small quantity of the liquid and precipitate, or of the precipitate alone after decanting the liquid if possible, and boil :—</td></tr>
<tr><td rowspan="2"><i>The precipitate dissolves:</i> to a portion of the NaHO solution add HCl in excess, brown SnS is reprecipitated.
Boil another portion of the precipitate after adding a little yellow Am_2S, then add HCl in excess, a yellow precipitate of SnS_2:—
<i>Presence of</i> Sn''
(Stannosum).
The presence of Sn'' should be confirmed by adding $HgCl_2$ to a part of the original HCl solution, a white precipitate shows <i>Presence of</i> Sn''.</td><td colspan="3"><i>The precipitate does not dissolve:</i> it may consist of CuS, HgS, Bi_2S_3, PbS.
<i>α. The original solution is blue or green</i>, and becomes intensely blue on addition of excess of AmHO:—<i>Presence of Cu</i> . Confirm by adding a few drops of H_2SO_4 to the original solution, and dipping into it a bright knife blade or piece of steel, a red film of Cu deposits.
<i>β. The original solution is colourless.</i> Test in separate portions for Hg, Bi, Pb by the following special tests:—</td></tr>
<tr><td>Immerse in the acid solution a bright strip of Cu; a grey film is slowly deposited, which becomes brilliant white when rubbed:—
<i>Presence of</i> Hg''
(Mercuricum.)
<i>Caution.</i>—A salt of Hg' may have been converted into an Hg'' salt during solution in HNO_3. The original substance should therefore be treated with AmHO; if it blackens Hg' is present, if not Hg'' was originally in the substance.</td><td>Pour another portion of the original solution into much cold distilled water; a white precipitate or milkiness:—
<i>Presence of Bi</i> .
<i>Note.</i>—If Bi is suspected and no milkiness appears, add excess of AmHO to the liquid ; Bi if present falls as hydrate ; filter, pour one or two drops of boiling HCl upon the precipitate, and let them drop through into large excess of water; a milkiness on stirring shows <i>presence of Bi</i> .</td><td>To a portion add H_2SO_4 a white precipitate:—
<i>Presence of Pb</i> .
The presence of Pb should be confirmed by adding to a part of the original solution AmHO in excess, then $H\bar{A}$ in excess, then K_2CrO_4 : a yellow precipitate shows <i>presence of Pb</i> .</td></tr>
<tr><td colspan="4">2. <i>The precipitate is yellow:</i> it may consist of CdS, As_2S_3, SnS_2 .
Boil the precipitate with NaHO in excess, after decanting the liquid if possible :—</td></tr>
<tr><td rowspan="2"><i>The precipitate does not dissolve :</i> it is bright yellow and pulverulent—
<i>Presence of Cd</i> .
The presence of Cd may be confirmed by showing that the precipitate produced by H_2S dissolves on being boiled with dilute H_2SO_4 .</td><td colspan="3"><i>The precipitate dissolves;</i> it may consist of As_2S_3 or SnS_2 ; examine portions of the original solution as directed below :—</td></tr>
<tr><td colspan="2">Acidify a portion with HCl, and place in it a piece of Zn resting on a piece of platinum foil : Sn is deposited on the Zn :—
<i>Presence of</i> Sn^{iv} (Stannicum).
The deposit of Sn should be detached from the Zn, and dissolved by boiling it in a test-tube with a little strong HCl, then $HgCl_2$ added ; a white precipitate confirms the <i>presence of</i> Sn^{iv}.</td><td>Acidify a portion with strong HCl, immerse in it some pieces of bright Cu and boil, a black film is deposited on the Cu :—
<i>Presence of As</i> .
<i>Note.</i>—If the yellow precipitate formed by H_2S appeared at once in the cold, the As is present as <i>Arsenosum ;</i> if it formed only on boiling, <i>Arsenicum</i> is present.</td></tr>
<tr><td colspan="4">3. <i>The precipitate is orange-red :</i> it may consist of Sb_2S_3 .
Confirm by acidifying a portion of the original solution with HCl, and immersing in it a piece of Zn resting on a slip of platinum foil, a black stain on the platinum (193) :—
<i>Presence of Sb.</i>
<i>Note.</i>—It occasionally happens, if the solution has not been sufficiently diluted, that H_2S gives a reddish precipitate with Pb-solution ; such a precipitate, however, becomes <i>black</i> on adding more H_2S-water.</td></tr>
</table>

346. TABLE III.A.—IRON GROUP.

The colour of the precipitate produced by addition of AmCl and AmHO will usually indicate whether it contains Fe, Al, Cr; the following tests may be made in confirmation.

To a portion of the original solution add KHO gradually until a precipitate has formed (see Note below Table), then add KHO in excess and stir well, one of the following results will be obtained :—

1. A *colourless* gelatinous precipitate is produced which dissolves in excess of KHO to a *colourless* solution: the precipitate reappears on gradual addition of HCl to the KHO solution :— *Presence of Al.*	2. A *pale-green* gelatinous precipitate forms, which dissolves in excess of KHO to a *green* solution; when PbO_2 is added to this green solution and the liquid is boiled and then acidified with $H\bar{A}$, a yellow precipitate is produced :— *Presence of Cr.*	3. A *reddish-brown* precipitate forms, insoluble in excess of KHO. Add KCyS to the original HCl solution, a blood-red coloration :— *Presence of* Fe'''.	4. A *dingy-green* precipitate forms, insoluble in excess of KHO, the precipitate if filtered off and exposed to the air gradually becomes brown :— *Presence of* Fe''.

Note.—if a colourless gelatinous precipitate has been produced by AmCl and AmHO, and is not produced by addition of KHO, a silicate is probably present. Its presence is confirmed by fusing the solid substance in a bead of $NaAmHPO_4$ (293); or by evaporating the acid solution to dryness, a residue insoluble in acid is SiO_2, and may be tested by the bead as directed above.

347. TABLE III.B.—ZINC GROUP.

The colour of the precipitate produced by addition of Am_2S or of H_2S to the alkaline solution will distinguish Zn or Mn from Ni, Co, and Fe, and will usually distinguish Zn and Mn also from one another. See *a* and *b* below :—

a. If the precipitate is light in colour, add to some of the original solution KHO gradually until a precipitate forms, then add KHO in excess. A *white* precipitate, dissolving in excess of KHO and reprecipitated by addition of H_2S proves *presence of Zn.* A *white or brownish* precipitate, insoluble in excess of KHO and gradually turning dark-brown when filtered off and exposed to the air shows *presence of Mn.*

b. If the precipitate is black it will consist of either CoS or NiS or possibly of FeS ; the distinction of the metals Ni and Co by the colour of their solutions—Ni being *green,* Co *pink*—and by the colours they impart to the borax bead is quite simple. FeS is only precipitated here from a *dilute* solution of a ferrous salt: Fe″ is best detected by (346).

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348. TABLE IV.—BARIUM GROUP.

Dip a loop of platinum wire into the original solution or into the HCl solution of the precipitate produced by Am_2CO_3, and hold the wire in the Bunsen flame. If the flame coloration thus obtained is not decisive, employ the tests placed at the foot of each column as confirmatory :—

A yellowish-green flame:– *Presence of Ba.*	*A crimson flame,* appearing deep red through the indigo-prism :— *Presence of Sr.*	*A red flame,* appearing dingy-green through the indigo-prism :— *Presence of Ca.*
Confirmatory.–Add $CaSO_4$ to a perfectly cold portion of the HCl solution, a precipitate forms *immediately.*	*Confirmatory.*–Add $CaSO_4$ to a perfectly cold portion of the HCl solution, no precipitate is produced, but a precipitate appears immediately on boiling.	*Confirmatory.*–Add $CaSO_4$ to part of the HCl solution and boil; no precipitate. To the rest add AmHO in excess, and $Am_2C_2O_4$: a white precipitate forms.
Note.—A brilliant green coloration may be produced by H_3BO_3.		

EXAMINATION FOR THE ACID-RADICLE.

349. If the acid-radicle has not been found already in the Preliminary Examinations (335–338*a*), or during the Examination for the Metal (341, 343) or elsewhere, it must be tested for as directed below. Since the solubility of the substance (332), and the metal it contains are known, much trouble is usually saved by referring to the "Table of Solubilities" (455–458). Thus a salt of the metal found, which is insoluble in water, cannot be present if the substance is soluble in water or is a neutral liquid : and an insoluble salt cannot be present in an acid solution or in a substance which is soluble in acids.

For the tests given below, use separate portions of the original substance : try the tests in succession until an acid-radicle is found.

350. Acidify a portion of the liquid, or if a solid dissolve it, with dilute HCl and add $BaCl_2$. If Pb, Hg′, or Ag has been found acidify with HNO_3 and add $Ba(NO_3)_2$, since HCl and chlorides would precipitate the above metals as chlorides. A white precipitate forms which does not disappear on boiling :—*Presence of a sulphate.*

351. Acidify a portion of the liquid, or if a solid is being examined dissolve the solid, with dilute HNO_3 ; filter off any precipitate or residue which may form, and add $AgNO_3$ solution : a precipitate forms, which does not disappear on warming. Note the colour of the precipitate ; and according as it is white, yellow, or orange, examine it by 1, 2, or 3 :—

<table>
<tr><td colspan="2">1. <i>The precipitate is white;</i> it may consist of AgCl, AgCy, Ag_4FeCy_6, or AgCyS. Make a portion of the original solution just acid with HCl, then add Fe_2Cl_6 :—</td></tr>
<tr><td>A deep blue precipitate :
<i>Presence of a ferrocyanide.</i></td><td>A blood-red coloration :
<i>Presence of a sulphocyanide.</i></td></tr>
<tr><td colspan="2">If Fe_2Cl_6 has yielded neither a precipitate nor coloration, indicating absence of a ferro- and sulpho-cyanide, decant the liquid from the precipitate caused by $AgNO_3$, pour upon the precipitate some strong HNO_3 and boil :</td></tr>
<tr><td>The precipitate remains undissolved :
<i>Presence of a chloride.</i></td><td>The precipitate dissolves :
<i>Presence of a cyanide.</i>
Confirm the presence of a cyanide in a portion of the original solution by boiling it with $FeSO_4$, Fe_2Cl_6, and KHO, and acidifying with HCl : a blue precipitate : <i>Presence of a cyanide.</i></td></tr>
<tr><td>2. <i>The precipitate is yellow</i>, and may consist of AgBr or AgI.
Test some of the original solution for an iodide by adding to it starch solution and KNO_2 solution, then acidifying (if not acid) with HCl : a deep blue coloration :
<i>Presence of an iodide.</i>
If an iodide is not thus detected, add to another portion of the original solution several drops of Cl water ; if the liquid becomes yellow, and on being shaken with CS_2 colours that liquid reddish-brown, it shows :
<i>Presence of a bromide.</i></td><td>3. <i>Orange-red precipitate.</i>
Make a portion of the original solution just acid with HCl, then add $FeSO_4$ solution ; a deep blue precipitate shows :
<i>Presence of a ferrocyanide.</i></td></tr>
</table>

Note.—If Hg has been found, $HgCy_2$ may be present, notwithstanding the formation of no precipitate with $AgNO_3$. If not already detected in (333 I.), refer to (306, *note* 2, p. 161).

352. Acidify some of the original liquid, or if it is a solid dissolve it, with dilute HNO_3; filter if necessary, add a small quantity of this acid solution to some $AmHMoO_4$ solution, stir well and warm gently:—

A yellow precipitate shows:— *Presence of a phosphate.*	*If no precipitate* forms heat to boiling, the formation of a yellow precipitate shows:— *Presence of an arsenate.*

The presence of a phosphate or arsenate may be confirmed by dissolving the yellow precipitate, separated by filtration or decantation, by pouring upon it a little AmHO; on adding to this solution AmCl and $MgSO_4$, a crystalline precipitate will form which is to be filtered off and washed with a little cold water: several drops of $AgNO_3$ solution are poured upon this precipitate on the filter; if it becomes *yellow* a phosphate is present, if *brown* an arsenate is present. The presence of an arsenate is further confirmed by a yellow precipitate (As_2S_3) appearing with H_2S when the liquid is boiled.

353. Acidify a portion of the original liquid, or if a solid stir it, with HCl; dip into this solution a piece of turmeric-paper, and dry the paper at a gentle heat; if the paper becomes *reddish-brown* when dry, and changes to *dingy-green* or *blue-black* when moistened with AmHO, the *presence of a borate* is shown.

354. The acid-radicles, *one* of which may still be present, are $''\overline{T}$, $''C_2O_4$ and $'F$. If the metal found belongs to Groups I., II., III., or IV., the examination for one of these radicles must be begun at (356), if the metal present is K, Na or NH_4 examine by (355).

355. Make the solution just alkaline with AmHO, then add $CaCl_2$ solution in some quantity, shake well and let stand if no precipitate forms at once: the preliminary examination (337) will usually have indicated which of these acid-radicles is present, and thus show according to which of the three columns below the precipitate is to be further tested:—

I. *A crystalline precipitate, appearing after some time*, shows probable *presence of a tartrate.*	II. *A white pulverulent precipitate* shows probable *presence of an oxalate.*	III. *A gelatinous flocculent precipitate* shows probable *presence of a fluoride.*
Confirm by 1 or 2 below:— 1. Decant the liquid from the precipitate, add a little water and a single drop of AmHO, drop in a small crystal of $AgNO_3$ and warm very carefully; a mirror is formed below the crystal, *presence of a tartrate.* This test may be tried also with the original solution by (322). 2. On acidifying some of the neutral original solution with $\overline{HA}$ and adding alcohol and $K\overline{A}$ (unless K or NH_4 has been detected in the substance), and stirring or shaking well, the formation of a crystalline precipitate shows *presence of a tartrate.*	The presence of an oxalate, already indicated by the test in (337, 5), must be confirmed either by warming some of the original substance with MnO_2 and H_2SO_4, and testing for CO_2 gas (319): or by the more tedious method of filtering off and drying some of the above precipitate of CaC_2O_4, then igniting it and testing for a carbonate with HCl (318 *a*).	Confirm by filtering and warming some of the precipitate, or of the substance if solid, in a small cup of lead or platinum with strong H_2SO_4; fumes will be evolved which etch a properly prepared watch glass (296).

356. An oxalate or fluoride of Ba, Sr, Ca, or Mg will have been already detected. A tartrate of any one of these metals is found by gently heating the finely powdered substance if it is solid, or the neutral solution if a liquid, with *very dilute* AmHO and a crystal of $AgNO_3$: the formation of a mirror shows *presence of a tartrate.* If a metal of Groups I., II., or III. has been found, the metal must be separated before testing for the acid-radicle.

If the metal belongs to Group I. or II., make the original solution just acid with HCl, and add H_2S-solution or pass H_2S (500 *a*) into the liquid for about five minutes; filter off the precipitate and add more H_2S to the filtrate, if no more precipitate forms*, boil in a porcelain dish until the liquid ceases to smell of H_2S, and examine by (355).

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* If any further precipitate forms add more of the reagent, filter, and test again by adding more of the reagent; this addition of reagent, filtration, and testing the filtrate is repeated until no further precipitate forms on addition of the reagent to the filtrate.

If the metal belongs to Group III. add AmCl, AmHO till alkaline, then Am_2S : boil and filter, add to the filtrate Am_2S if no more precipitate forms (see footnote, page 188), add $H\bar{A}$ in excess, boil, filter off S if necessary, and examine by (355).

Note.—If no acid-radicle can be found in any quantity and the substance is not metallic in appearance, it is probably an oxide or a sulphide; most sulphides yield a sulphate or sulphuric acid when boiled with strong HNO_3, which may be detected after dilution by (350); an oxide may often be known by its colour (see footnote, page 170) or by some other special property or test, *e.g.* MnO_2 by evolving Cl when heated with HCl, Pb_3O_4 by leaving brown PbO_2 when treated with dilute HNO_3.

EXAMINATION OF GROUP III.A.

357. The precipitate formed by addition of AmCl and AmHO to the solution of a substance insoluble in water may consist of Al_2Ho_6, Fe_2Ho_6, Cr_2Ho_6; or of Fe, Al, Cr, Zn, Mn, Ni, Co, Ba, Sr, Ca, Mg as phosphate or oxalate; or of Ba, Sr, Ca as chromate or fluoride. The systematic examination of a precipitate in Group III.A for these substances is a difficult matter; such an examination is however only necessary, when an oxalate (337, 5), a chromate (343) or a fluoride (338) have been already detected, or when a phosphate is found. If a precipitate forms in Group III.A, and the above acid-radicles are absent, proceed as directed in (346): if the above acid-radicles are present, the metal, unless certainly detected during the Preliminary Examination, must be tested for as directed below.

358. Note the appearance of the precipitate caused by AmCl and AmHO, then add Am_2S to the liquid and precipitate, stir well and note again the appearance of the precipitate: examine further by 1 or 2 (page 190).

1. *The precipitate after addition of Am_2S is black*, it may consist of FeS, CoS, or NiS; the means of distinguishing these three metals is given below. Note first the colour of the original solution (334, 1).

<table>
<tr>
<td rowspan="2">a. <i>The colour of the original solution was yellow or reddish-brown</i> :—
<i>Presence of Fe'''.</i>
(<i>Ferricum.</i>)
This is confirmed by the precipitate produced by AmCl and AmHO in the original solution having been <i>pale-yellow or reddish-brown</i>, and becoming black on addition of Am_2S.
As a confirmatory test add to a portion of the original solution, acidified with HCl if necessary, a few drops of KCyS solution, a <i>blood-red</i> coloration is produced.</td>
<td rowspan="2">b. <i>The colour of the original solution was pink</i>, or possibly blue, becoming pink on dilution: <i>Presence of Co.</i>
The presence of Co may be confirmed by adding excess of KHO to the original solution, a blue precipitate turning red on boiling shows <i>the presence of Co.</i>
Co will also have been detected by its borax bead (333, IV.)</td>
<td colspan="2">c. <i>The colour of the original solution was green</i> :—<i>Presence of Ni or Fe''.</i>
Note the effect which was produced by the addition of AmCl and AmHO :—</td>
</tr>
<tr>
<td><i>A blue solution was produced</i> :—<i>Presence of Ni.</i>
The presence of Ni may be confirmed by adding excess of KHO to a part of the original solution, a light green precipitate shows <i>the presence of Ni.</i>
Ni will also have been detected by its borax bead (333, IV.)</td>
<td><i>A dingy-green precipitate</i>, which, when shaken round in a porcelain dish and left for several minutes exposed to the air, becomes brown :—<i>Presence of Fe''.</i>
(Ferrosum.)
Confirm by adding K_6Cfy_2 to some of the original solution, acidified with HCl if necessary; a dark blue precipitate is produced.</td>
</tr>
</table>

2. *The precipitate, after addition of Am_2S, is white or light-coloured.*—It may consist of Cr_2Ho_6, Al_2Ho_6, ZnS, MnS; [or of $BaCrO_4$, or Ba, Sr, Ca, Mg, as phosphate, oxalate, fluoride, silicate, or possibly borate]. The substances in brackets can however only be present if the liquid given for analysis was acid in reaction, or if a solid was given which was insoluble in water.

To a small portion of the original solution add KHO drop by drop until a precipitate is produced. (See Note below the Table.) Note the colour and appearance of this precipitate, then add a larger quantity of KHO and stir or shake well; one of the following results (*a* or *b*) will be noticed:—

a. *The precipitate at first produced by KHO re-dissolves when the KHO is added in larger quantity;* indicating the presence of Al, Zn, or Cr. Note the appearance of the precipitate:—

A pale green flocculent precipitate:— *Presence of Cr.* Confirm by boiling some of the green KHO solution with lead-peroxide (PbO_2), a yellow liquid is obtained, which, if decanted, yields a yellow precipitate on addition of $H\bar{A}$ in excess. The colour of the solution and of the borax bead will also have detected Cr. *Note.*—If the Cr was present as a chromate, AmCl and AmHO will have yielded no precipitate; but there will have been a green precipitate on addition of Am_2S and heating.	*A white precipitate, which is re-precipitated from the KHO solution,* by addition of a few drops of H_2S-water but is not re-precipitated by addition of AmCl: *Presence of Zn.* Confirmed by no precipitate having been produced by AmCl and AmHO, but a white precipitate by addition of Am_2S, or of H_2S after AmHO.	*A white flocculent precipitate, not reprecipitated* from the KHO solution by addition of a few drops of H_2S-water or by boiling, but reprecipitated immediately if sufficient AmCl solution is added:— *Presence of Al* (see Note). *Note.*—$AlPO_4$ is precipitated here also, and is soluble in KHO, but differs from Al_2Ho_6 in being reprecipitated from the KHO solution by addition of excess of $H\bar{A}$; if PO_4 has been detected the analysis is finished. $AlPO_4$ is soluble only in acids, hence the original liquid must' have been acid or the original solid substance insoluble in water.

Note.—Sometimes a flocculent precipitate is produced on adding AmCl and AmHO to the original solution, and yet no precipitate is produced by KHO; this renders probable the presence of an alkaline silicate. In this case evaporate a portion of the solution to dryness with HCl; warm the residue with HCl: if any insoluble residue is left, the *presence of a silicate* is proved; filter and test the filtrate for K or Na.

b. *The precipitate at first produced by KHO does not redissolve on addition of more KHO;* the precipitate will have one of the following appearances:—

A white or dingy-yellow precipitate, rapidly darkening when shaken in the air:— *Presence of Mn.* *Confirmed* in the preliminary examination by producing a green mass when fused with Na_2CO_3 and KNO_3, and by no precipitate having been produced on addition of AmCl and AmHO, unless the solution was exposed for some time to the air.	*A pale yellow precipitate, not darkening in the air:* this precipitate will also have been produced by AmCl and AmHO and its colour will not have been altered by addition of Am_2S:— *Presence of $BaCrO_4$.* Ba has been already *confirmed* by the flame coloration (333, II.), and a chromate by the change of colour of the orange-red liquid to green on adding H_2S (343).	*A white precipitate, not darkening in the air:—* Presence of Ba, Sr, Ca, or Mg as phosphate, oxalate, &c. Pass on to (359). *Note.*—This precipitate can only be formed if the original solution was acid.

359. *The precipitate may consist of Ba, Sr, Ca, or Mg as phosphate, oxalate, borate*, fluoride, or silicate.*

Examine for the metal and acid-radicle as directed below, working through the columns from left to right, unless the presence of a phosphate (339), oxalate (333, I. 4, or 337, 5), borate (334, V., or 333, II., or 338 *a*), or fluoride (338), has been already proved, in which case proceed at once to examine according to (360), or by column 1, or 2, or 3 below.

If this precipitate is proved to contain a phosphate, oxalate, borate, fluoride, or silicate, the further examination for an acid-radicle becomes unnecessary.

1. Add AmHO in excess to a portion of the original solution†, filter, dry the precipitate and heat a portion of it to a dull red heat for several minutes on platinum foil. Place the foil in a test-tube and pour a few drops of dilute HCl upon it; if the residue dissolves with effervescence, it shows the *presence of an oxalate,* Test this HCl solution for Ba, Sr, Ca, Mg, by (339), using it as the original solution. If the ignited precipitate does not dissolve with effervescence, pass on to column 2.	2. Stir some of the precipitate produced by AmHO†, column 1, on a watch glass, with a little water to which a few drops of HCl have been added, until the precipitate is dissolved. Dip into the liquid a slip of turmeric paper, and dry at steam heat; the slip appears *reddish-brown*, and becomes *dingy-green* when moistened with AmHO:— *Presence of a borate.* Examine for the metal present by (361). If a borate is not found, proceed to column 3.	3. Place another portion of the precipitate produced by AmHO†, column 1, in a leaden cup or platinum crucible, add strong H_2SO_4 and warm gently, covering the vessel with a watch glass, which has been coated with a film of wax and has had characters traced with a sharp point through the wax (296); the glass is etched:— *Presence of a fluoride.* Examine for the metal present by (361). If no fluoride is found, proceed to column 4.	4. If phosphate oxalate, borate and fluoride have been proved to be absent, a silicate is probably present. Test for it par. 361.

360. *Examination of a phosphate—Explanation of Method.*—In order to detect Ba, Sr, Ca, or Mg present as phosphates, the PO_4

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* Borates of Ba, Sr, and Ca, are rarely precipitated in Group III., since they are soluble in the AmCl formed on adding AmHO to the HCl solution.

† If a solid is being examined, a portion of the original solid is employed for this test, instead of the precipitate produced by AmHO in the HCl solution.

must be removed from the solution and the metal left as chloride. This is effected by neutralising all HCl and leaving the solution acidified only by free $H\bar{A}$; by *gradual* addition of Fe_2Cl_6 and boiling, all PO_4 is precipitated as $FePO_4$ which though soluble in HCl is perfectly insoluble in $H\bar{A}$. On filtering, $FePO_4$ remains upon the filter, and the filtrate contains the metal (Ba, Sr, Ca, or Mg) as chloride, and is perfectly free from phosphate.

Method.—Add to a portion of the cold original solution AmHO drop by drop, whilst constantly shaking the liquid, until a slight precipitate is formed which does not disappear by shaking; then add $H\bar{A}$ and $Na\bar{A}$ solution, and drop in Fe_2Cl_6 until the liquid after being well shaken has a brown or red colour; boil and filter immediately. The precipitate is thrown away. To the filtrate AmCl is added, then AmHO until it is just in excess, boil and filter; the liquid or filtrate, which must smell strongly of NH_3, is then tested for Ba, Sr, Ca, and Mg by (339), this liquid taking the place of the original solution.

If any precipitate is formed by Am_2CO_3, it must be well washed several times with boiling water to remove Na, since else the intense yellow coloration of Na would mask that of Ba, Sr, or Ca.

361. A portion of the original substance is placed in an evaporating basin, which is then nearly filled with dilute HCl, and the liquid is entirely boiled away ; the dish is once more filled with acid, and again evaporated to dryness : the residue in the dish is then warmed with dilute HCl, and filtered if any residue is left :—

Solution : the HCl solution is examined for Ba, Sr, Ca, Mg by (339), this solution being treated as is there directed for the original solution.	*Residue :* if any residue remains insoluble in HCl, it proves :— *Presence of a silicate.*

The evaporation to dryness with HCl removes H_3BO_3 and HF, which volatilise, whilst SiO_2 is left insoluble in HCl and therefore remains as an insoluble powder on afterwards warming with HCl ; this residue of SiO_2 also remains, however, if HF was present, being caused by the corrosion of the dish ; since however HF if present has already been found, no notice will then be taken of the residue.

THE SUBSTANCE POSSESSES METALLIC LUSTRE.

366. This shows the probable absence of an acid-radicle. The only non-metallic elements likely to be present are free carbon as graphite, free iodine, or combined sulphur, since several sulphides are decidedly metallic in appearance*. The presence of the metals Ni, Co, Fe in the free state, and of Fe_3O_4, may be shown by the substance being attracted by a magnet. Pb, or graphite, will be recognised by marking paper when rubbed upon it, as a black-lead pencil does: the mark due to Pb disappears if wetted with dilute HNO_3.

PRELIMINARY EXAMINATION.

Experiment.	Observation.	Inference.
I. Heat a portion of the substance in a small ignition-tube (10).	1. A black lustrous mirror forms	Presence of As.
	2. A yellow sublimate forms, melting, when heated, to brown drops	Presence of S.
	3. Violet vapour is given off, condensing to black scales on the sides of the tube	Presence of I.
	If the substance sublimes entirely in violet vapours	Iodine alone is present.
II. Heat a portion in the inner blowpipe flame in a cavity scooped on a piece of wood-charcoal.	1. Smell of garlic	Presence of As.
	2. If an incrustation forms, refer to par. (333, V.) where in the second and third column will be found the appearance of the incrustation and the metal whose presence it indicates.	
	3. Smell of burning sulphur	Presence of S.
	4. A green flame coloration	Presence of Cu.
III. Place a portion of the substance in a piece of hard glass tube open at both ends, and heat it strongly in the Bunsen flame, and afterwards in the blowpipe flame if necessary.	1. A gas is evolved from the upper end which smells of burning sulphur and turns a piece of filter-paper moistened with $K_2Cr_2O_7$ solution green	Presence of S.
	2. The substance burns completely but slowly away, glowing when strongly heated. This effect is best seen by heating it strongly on Pt-foil by directing the blowpipe flame on the under surface of the foil	Presence of C.

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* The most common sulphides which possess metallic lustre are "copper pyrites," and "iron pyrites," which resemble brass in appearance: and "galena," resembling lead.

After completing the preliminary examination proceed as directed under (332).

No acid-radicle need be tested for except S: a portion of the substance is dissolved in strong HNO_3, and after diluting with water H_2SO_4 is tested for by $BaCl_2$: if a white precipitate forms insoluble on boiling, it confirms the *presence of S*.

THE SUBSTANCE IS INSOLUBLE BOTH IN WATER AND IN ACIDS.

367. It may consist of any one of the following substances, those included in brackets being less likely to occur than the others, since they are soluble in sufficient water or acid on heating. As will be seen below the colour serves to give some notion as to what substance is present.

1. *White:* $BaSO_4$, $SrSO_4$, ($CaSO_4$), $PbSO_4$, $(PbCl)_2$, AgCl*, SiO_2, (Al_2O_3 ignited), SnO_2, Sb_2O_5, Sb_2O_4, CaF_2.
2. *Dark-coloured:* ignited Fe_2O_3 (dark-brown or black), ignited Cr_2O_3 (dark-green), $FeCr_2O_4$ and ignited $PbCrO_4$ (dark-brown), C (black).
3. *Yellow:* S, AgBr*, AgI*, (PbI_2).

An insoluble silicate may also be present.

For a fuller description of these substances see (475).

According to the colour of the substance examine it by (368 or 369).

368. *The substance is white.* If sufficient substance is at hand try the following preliminary tests, if not, proceed at once to (368 *a*). Pour upon a small portion a drop of Am_2S; if it blackens the presence of Pb or Ag is probable, if it becomes yellow or orange-red the presence of Sn or Sb is probable; pass to test III. in the following Table.

If no change of colour is produced by Am_2S, try tests I and II.

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* Darkens quickly in sunlight, and slowly in ordinary daylight.

Experiment.	Observation.	Inference.
I. Moisten a clean loop of platinum wire, immerse it in the powdered substance, and heat the adhering powder for a short time strongly in the *inner* blowpipe flame; moisten the loop with a drop of strong HCl, and hold it in the Bunsen flame	A *yellowish-green* flame.	*Presence of Ba.* } Test for SO_4 by Exp. II.
	A *crimson* flame, appearing *deep red* through the indigo-prism.	*Presence of Sr.* } Test for SO_4 by Exp. II.
	A *reddish* flame, appearing *dusky green* through the indigo-prism.	*Presence of Ca.* } Test for SO_4 by Exp. II.
II. Fuse a portion of the substance, mixed with Na_2CO_3 in fine powder, in a cavity scooped upon a piece of wood-charcoal, in the inner blowpipe flame, *produced from a spirit-lamp flame.*	The cooled mass, if detached from the charcoal, placed upon a bright silver coin, and moistened with water, gives, when crushed with the blade of a knife, a black stain.	*Presence of a sulphate.* *Note.*—If SO_4 is not found, test for F by (338 or 296), or examine the substance by (368 *a*).
III. Mix some of the finely powdered substance with powdered Na_2CO_3 and KCy, on a small cavity made in a piece of wood-charcoal, and heat the mixture in the inner blowpipe flame. If metallic globules form, detach one and strike it smartly with the pestle on the inverted mortar. Also take a globule on the point of a penknife and try if it marks paper as black lead does.	White metallic *malleable* globules, which mark paper, and if dissolved in HNO_3 give a white precipitate with H_2SO_4; a yellow incrustation is also formed on the charcoal.	*Presence of Pb* as chloride, sulphate, or chromate. Refer to the *Note* below this Table.
	White metallic *malleable* globules which do not mark paper; they dissolve in HCl, the solution giving a white precipitate with $HgCl_2$.	
	White metallic *brittle* globules, which, if dissolved in boiling HCl, give an orange-red precipitate with H_2S: white incrustation.	*Presence of SnO_2.*
	White scales, no incrustation: the metal is insoluble in HCl, and if dissolved in HNO_3 gives no precipitate with H_2SO_4 but a white precipitate with HCl; the original substance is instantly blackened by a drop of Am_2S, and is dissolved on being warmed with AmHO.	*Presence of Sb_2O_4 or Sb_2O_5.* *Presence of AgCl.* [See also (368*a*).]

Note.—Boil some of the substance with much water, add HNO_3 and $AgNO_3$, a white precipitate shows *presence of $PbCl_2$.*

Test for chromate by fusing in a porcelain crucible with $Na_2CO_3 + K_2CO_3$; a yellow mass on cooling shows *presence of $PbCrO_4$.*

The presence of $PbSO_4$ may be proved by test II., and by pouring upon the original substance $\bar{H}\bar{A}$ then excess of AmHO, and boiling :-

the substance will dissolve : acidify a portion of the solution with $H\bar{A}$, and add K_2CrO_4, a yellow precipitate : *Presence of Pb.* Acidify another portion with HCl, and add $BaCl_2$ a white precipitate insoluble on boiling.—*Presence of SO_4.*

If none of the substances mentioned in the above Table have been detected, test for Al_2O_3 by heating the substance strongly on charcoal in the outer blowpipe flame after moistening it with $Co(NO_3)_2$ solution, a blue mass shows *presence of Al_2O_3.*

If Al_2O_3 is not found fuse some of the substance in a clear bead of $NaAmHPO_4$, it floats undissolved :—*Presence of SiO_2*, or a silicate. Proceed to (368 *a*).

368 *a.* Fuse a portion of the finely powdered substance, mixed with two or three times as much fusion mixture, for several minutes on a piece of platinum foil (or if Pb or Ag is present, in a porcelain crucible) in the blowpipe flame; boil the cool mass with water, filter and wash the residue.

Examination of the undissolved residue.—Heat the residue with dilute HNO_3, if it is not dissolved in a short time, decant and keep the HNO_3, and heat the residue with a small quantity of dilute HNO_3 and HCl mixed : if the residue is still undissolved it has been insufficiently fused.

The acid solution is then examined for the metal by (339).

Examination of the water solution.—When the metal present has been detected, it will usually be possible to limit the number of acid-radicles which need be tested for by referring to the list in (367). Tests are given below for all acid-radicles likely to occur in insoluble substances. Separate portions of the water solution are to be used.

Sulphate: acidify a portion with HCl and add $BaCl_2$: a white precipitate.

Chloride: acidify a portion with HNO_3 and add $AgNO_3$, a white precipitate easily soluble in AmHO. If the precipitate has a yellow tinge and is not easily soluble in AmHO, add to a fresh portion of the water solution dilute Cl-water drop by drop, shaking well between each addition, a brown coloration of the CS_2 shows a *bromide*, a violet coloration an *iodide.*

Silicate: make a portion acid with HCl and evaporate to perfect dryness, if on warming with dilute HCl an insoluble residue is left, this proves the presence of silica or a silicate.

Fluoride: acidify with $H\bar{A}$ and add $CaCl_2$, a gelatinous white precipitate forms. Confirm by tests (338 or 296) tried on some of the original substance.

369. *The substance is dark-coloured.* (See 367, 2.)

Place some of the substance on a piece of platinum foil and heat the foil strongly on its under surface by the blowpipe flame :—

The substance burns away slowly but completely :— *Presence of C.*	*The substance does not burn away;* place upon it three or four times as much powdered Na_2CO_3 and KNO_3, and fuse for some time :—
	1. The substance dissolves, forming a yellow mass when cold :—*Presence of Cr_2O_3.* 2. The substance remains undissolved as a dark brown powder, and the mass on cooling is white :—*Presence of Fe_2O_3*.* Confirm by boiling the mass with water, filtering and fusing the brown residue into a borax bead (100).

The substance is yellow (367, 3).

Heat a portion of it strongly in a small ignition-tube; one of the following results will occur :—

It fuses and sublimes, the sublimate being yellow and melting to reddish-brown drops when heated :— *Presence of S.* *Note.*—If the substance sublimes entirely it consists only of sulphur.	*It fuses but does not sublime.* Examine a portion of the substance for Ag and Pb and for Br and I by (368 *a*).

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* $FeCr_2O_4$ shows both reactions 1 and 2, since it contains both Fe and Cr.

ANALYSES OF SIMPLE SALTS, SHOWING HOW TO ENTER RESULTS.

A. *Liquid given for analysis.*

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Noted the colour of the liquid.	Blue.	Presence of Cu.
2. Dipped into it a piece of blue litmus-paper.	The paper was turned red, but on addition of Na_2CO_3 no effervescence occurred.	Presence of a salt with acid reaction.
3. Evaporated a few drops slowly upon a watch glass.	Pale blue residue left.	Presence of some dissolved solid.
Ignited the residue strongly.	Blackened without smell of burning.	Absence of organic acid-radicles.
4. Added KHO solution and boiled.	No smell of NH_3.	Absence of NH_4.
5. Dipped a loop of platinum wire into the solution and held it in the flame.	*Bright green* flame; on moistening the wire with strong HCl and again holding it in the flame it gave a blue coloration.	Presence of Cu.
6. Dipped a clear borax bead into a portion of the residue from 3, and fused in the outer and inner blowpipe flame.	In outer flame the bead was *green* whilst hot, *blue* when cold. In inner flame it became colourless.	Presence of Cu.

Preliminary Examination for the Acid-radicle.

Experiment.	Observation.	Inference.
1. Added dilute H_2SO_4 and warmed.	No gas was evolved.	Absence of carbonate, sulphite, sulphide, and nitrite.
2. Added strong H_2SO_4 and warmed.	No gas was evolved. And no red fumes on adding Cu clippings.	Absence of chlorate. Absence of nitrate.

Examination for the Metal.

To a portion of the liquid added HCl:—

No precipitate. Absence of Group I.	Added strong H_2S-water:—
	A brownish-black precipitate. Presence of Sn″, Hg″, Bi, Pb, or Cu. Allowed the precipitate to settle, poured off as much of the liquid as possible and boiled the precipitate with excess of NaHO, it remained undissolved. Since the original solution was blue, a clean knife blade was dipped into a portion of it acidified with H_2SO_4, red copper was deposited on the steel:— *Presence of Cu.*

Examination for the Acid-radicle.

Added HCl and $BaCl_2$; a white precipitate was produced which did not disappear on boiling:—*Presence of sulphate.*

Found Cu, SO_4.

B. *Liquid given for analysis.*

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Noted the colour of the liquid.	Light yellow.	Presence of a neutral chromate.
2. Dipped red litmus-paper into the liquid.	It was turned *faintly* blue.	Probable presence of an alkali-salt.
3. Evaporated a few drops to dryness upon a watch glass.	A yellow residue remained.	Presence of some dissolved solid.
Ignited the residue strongly.	No blackening.	Absence of organic acid-radicle.
4. Boiled a portion with excess of KHO.	No smell of NH_3.	Absence of NH_4.
5. Dipped a loop of platinum wire into the solution, and held it in the Bunsen flame.	A pale violet flame, appearing crimson through the indigo-prism.	Presence of K.
6. Dipped a clear borax-bead into the residue from 3, and fused in the outer and inner blowpipe flames.	Outer flame: *brown*, hot; *green*, cold. Inner flame: *green*, hot and cold. *Brown* colour not reappearing in the outer flame.	Presence of Cr as a chromate.

Examination for the Metal.

Since the liquid was alkaline in reaction, added to a small portion of it a few drops of HNO_3 until it became acid, this produced no precipitate.

To another portion added HCl, the liquid turned from yellow to orange-red, indicating presence of a neutral chromate:—

<table>
<tr><td rowspan="4"><i>No precipitate.</i> Absence of Group I.</td><td colspan="4">Then added H_2S-water and warmed :</td></tr>
<tr><td rowspan="3">The liquid became green, and white S was deposited :
<i>Presence of a chromate.</i></td><td colspan="3">To another portion of the original solution added AmCl, then AmHO, no precipitate formed : then added Am_2S and boiled :</td></tr>
<tr><td rowspan="2"><i>No precipitate</i> in the cold, but on boiling a green flocculent precipitate gradually formed, evidently consisting of Cr_2Ho_6 reduced from the chromate.</td><td colspan="2">Added to another portion of the ½ solution AmCl, AmHO, and Am_2CO_3 :</td></tr>
<tr><td><i>No precipitate.</i> Absence of Group IV.</td><td>Added to the same portion Na_2HPO_4 and stirred :
<i>No precipitate. Absence of Mg.</i></td></tr>
</table>

Since K was found by the flame coloration in the preliminary examination, its presence was confirmed by stirring a portion of the solution, to which a few drops of HCl had been added, with $PtCl_4$ on a watch glass; a yellow crystalline precipitate forming on the lines rubbed by the rod:—*Presence of K.*

Found K, CrO_4.

C. A *white crystalline substance given for analysis.*

Boiled a small portion with water; it dissolved completely.

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Heated in small dry test-tube.	The substance gave off red fumes and oxygen gas, which inflamed a glowing splinter of wood. It left a yellow residue.	Presence of a nitrate of a heavy metal. Presence of Pb, Sn, or Bi.
2. Dipped a loop of moistened platinum wire into the powdered substance, moistened with strong HCl and held in the Bunsen flame.	A pale blue flame.	Presence of As, Sb, Pb.
3. Heated a small portion in a cavity on wood-charcoal in the inner blowpipe flame.	Deflagration occurred. A white malleable globule remained which easily marked paper. A yellow incrustation.	Presence of a nitrate or chlorate. Presence of Pb.
4. Fused on charcoal in the inner blowpipe flame with Na_2CO_3 and KCy.	Same result as with 3.	Presence of Pb.

Examination for the Metal in Solution.

Boiled a portion of the powdered substance with water, cooled, added HCl to a part of the clear solution, a white precipitate was formed, which disappeared on boiling, but appeared again in the crystalline form on cooling the liquid (*probable presence of Pb*).

Confirmed the presence of Pb by decanting the liquid and boiling the precipitate with AmHO, it remained white and did not dissolve; decanted the ammoniacal liquid, dissolved the precipitate in a little $H\bar{A}$, and added K_2CrO_4, a yellow precipitate:—*Presence of Pb*.

Since a nitrate was found in the preliminary examination, its presence was confirmed by adding strong H_2SO_4 to the aqueous solution of the substance, and pouring solution of $FeSO_4$ carefully upon the cooled liquid in a test-tube; a brown ring formed on the surface of the acid:—*Presence of* NO_3.

Also some of the solution when heated with Cu and strong H_2SO_4, evolved reddish-brown fumes:—*Presence of* NO_3.

Found:—Pb, NO_3.

D. *A white powder, emitting no smell, given for analysis.*

Boiled some of the powdered substance with water, it did not dissolve: added a few drops of strong HCl and boiled, the substance dissolved completely (absence of Group I.) without effervescence.

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Heated in a small dry test-tube.	No change.	Absence of volatile and fusible substances and of water of crystallisation, &c.
2. Dipped a moistened loop of platinum wire into the powdered substance, moistened the powder with strong HCl, and held it in the Bunsen flame.	No flame coloration.	Probable absence of K, Na, Ba, Sr, Ca, Cu, &c.
3. Heated a portion of the powder strongly on charcoal in the inner blowpipe flame.	The substance did not fuse.	Absence of alkali salt.
	A white luminous residue, which when moistened on red litmus-paper showed an alkaline reaction.	Presence of Ba, Sr, Ca, Mg.

Examination of the solution.

To a portion of the HCl solution added H_2S-water; no precipitate:—*Absence of Group II.*

To another portion of the HCl solution added AmCl and AmHO in excess, a white flocculent precipitate: then added Am_2S, the colour of the precipitate was unchanged.

To a few drops of the HCl solution added KHO drop by drop, until a white flocculent precipitate formed, then added excess of KHO and boiled, the precipitate did not dissolve, and it did not become discoloured in the air:—*Presence of Ba, Sr, Ca, Mg as phosphate, oxalate, &c.*

Added a few drops of the original HCl solution to some $AmHMoO_4$ solution in a test-tube, and warmed gently, a yellow precipitate:—*Presence of PO_4.*

To another portion of the *cold* HCl solution added AmHO drop by drop until a precipitate formed which did not dissolve when shaken, then added $H\bar{A}$ and $Na\bar{A}$ solution until this precipitate dissolved when the liquid was well shaken. Fe_2Cl_6 was added until the liquid appeared red; it was then boiled and immediately filtered. To the filtrate AmHO was added in excess and the precipitate filtered off, then Am_2CO_3 was added, a white precipitate formed:—*Presence of Ba, Sr,* or *Ca*.

Filtered and dissolved the precipitate by pouring upon it a few drops of boiling HCl; dipped a loop of platinum wire into the solution, and held it in the Bunsen flame, a reddish flame appearing dingy-green through the indigo-prism:—*Presence of Ca.*

A few drops of the solution boiled with $CaSO_4$ gave no precipitate:—absence of Ba and Sr, therefore the presence of Ca was confirmed by adding to the rest of the solution excess of AmHO, then $Am_2C_2O_4$, a white precipitate:—*Presence of Ca.*

Found Ca, PO_4.

E. *A white odourless powder given for analysis.*

Boiled a portion of the substance with water, it did not dissolve: added HCl and heated again, it dissolved completely (absence of Group I.) without effervescence.

Preliminary Examination.

Experiment.	**Observation.**	**Inference.**
1. Heated a portion in a small dry test-tube.	**The substance blackened slightly; it evolved CO_2 which turned a drop of lime-water milky. The residue in the test-tube effervesced with HCl whereas the original substance did not.**	**Presence of an organic salt of K, Na, Ba, Sr, Ca, or Mg.**
2. Held a portion of the substance on a loop of platinum wire in the Bunsen flame; then moistened with HCl and again held in the flame.	**A *red* coloration, appearing *dusky-green* through the indigo-prism.**	**Presence of Ca.**
3. Heated on charcoal in inner blowpipe flame.	**A white luminous mass remained, which when moistened on red litmus paper turned it blue.**	**Presence of Ba, Sr, Ca, or Mg.**

Examination for the Metal.

Added to a portion of the HCl solution strong H_2S-water.

No precipitate:— Absence of Group II.	To another portion of the HCl solution added AmCl then AmHO in excess, *a white precipitate formed;* added Am_2S, the colour of the precipitate remained unaltered. To another portion of the solution added KHO drop by drop until a precipitate formed, then more KHO and boiled; a white precipitate insoluble in KHO, and not darkening in the air; —*Presence of oxalate, phosphate, &c., of alkaline earths.* Since the presence of an oxalate of an alkali- or alkaline earth-metal was indicated in the preliminary examination, a portion of the original substance was heated on platinum foil, then dissolved in HCl (effervescence) and to the solution excess of AmHO and of Am_2CO_3 was added. The liquid was warmed and put aside till the precipitate subsided, then the liquid was decanted and the precipitate dissolved in a few drops of HCl. A platinum wire dipped into this HCl solution gave a *red* flame coloration, appearing *dusky-green* through the indigo-prism:—*Presence of Ca.* [Confirmed by another portion of this HCl solution giving a white precipitate with excess of AmHO and $Am_2C_2O_4$.]

The presence of C_2O_4 was confirmed by heating some of the original substance with strong H_2SO_4: gases were evolved which rendered milky a drop of lime-water, and burnt with a blue flame, showing presence of CO_2 and CO :—*Presence of* C_2O_4.

Found Ca, C_2O_4.

F. *A yellow metallic-looking substance given for analysis.*

Preliminary Examination.

Experiment.	Observation.	Inference.
1. Heated a portion of the substance strongly in a small tube closed at one end.	A yellow sublimate formed which melted into brown drops: a smell of burning S was evolved, and a piece of paper dipped into $K_2Cr_2O_7$ solution when placed in the mouth of the tube became green. A brown residue left.	Presence of S. Probable presence of Fe.
2. Heated strongly on charcoal in the inner blowpipe flame.	Strong smell of burning S. The residue fused into a dark-coloured globule, which when cold was strongly attracted by the magnet.	Presence of S. Probable presence of Fe
3. Heated a fragment strongly in a glass tube open at both ends, and held obliquely in the flame.	A gas was evolved with a suffocating smell, and which turned $K_2Cr_2O_7$ paper green.	SO_2 from combustion of S.

Solution of substance.

Warmed the powdered substance with a mixture of strong HNO_3 and HCl, it dissolved completely; boiled down in an evaporating dish, adding strong HCl several times; when nearly dry, diluted with water.

Examination of the solution for the Metal.

Since the substance dissolved entirely in HCl, Group I. is absent; to a portion of the solution added H_2S-water :—

The solution became colourless, and white S was precipitated :— *Probable presence of a ferric salt.*	To another part of the solution added AmCl, then excess of AmHO, a brown flocculent precipitate formed, *presence of* Fe''': then added Am_2S, and heated : the precipitate became black, and on settling left a *yellow* solution :—*Absence of* Ni. Confirmed the presence of Fe by adding to a fresh part of the original solution K_4FeCy_6 solution, *a dark blue precipitate : Presence of* Fe'''.

Confirmed the presence of S by adding to a part of the original solution $BaCl_2$, a white precipitate formed which did not disappear on boiling :—*Presence of* SO_4 *derived from oxidation of sulphur.*

Found Fe, S.

G. *A white powder given for analysis.*

Boiled a small portion with water, it did not dissolve.

Boiled another portion with HCl, it did not dissolve : added HNO_3 and heated again, the substance was not dissolved. Hence the substance is insoluble in water and in acids, and since it is white, it can only be $BaSO_4$, $SrSO_4$, ($CaSO_4$), $PbSO_4$, ($PbCl_2$), AgCl, SiO_2, Al_2O_3, SnO_2, Sb_2O_4, or CaF_2.

Poured Am_2S upon some of the substance : its colour was unchanged, *probable absence of Pb, Ag, Sn, Sb.*

Preliminary Examination.

Experiment.	Observation.	Inference.
1. Took some of the powder up with a loop of moist platinum wire, ignited it strongly in the inner blowpipe flame, moistened it with HCl, and held in the Bunsen flame.	A yellowish-green coloration.	Presence of Ba.
2. Fused a portion with Na_2CO_3 on charcoal in the inner blowpipe flame, placed the cooled mass on a bright shilling, and moistened it with a drop of water.	A black stain on the coin.	Presence of S.

Examination of the solution.

Fused some of the powder with fusion mixture on platinum foil: boiled the cold mass with water, filtered.

Filtrate, made acid with HCl and added $BaCl_2$, a white precipitate, which did not disappear on boiling the liquid:—*Presence of SO_4*.

The residue on the filter was well washed with boiling water, and boiling dilute HCl poured upon it, it dissolved completely:—

Absence of Group I.	To a portion added strong H_2S-water and heated:		
	No precipitate. Absence of Group II.	To another portion added AmCl, then AmHO in excess, then Am_2S, and boiled:	
		No precipitate. Absence of Group III.	To another portion of the original solution added AmCl, AmHO, and Am_2CO_3:
			A white precipitate: Presence of Ba, Sr, or Ca. A platinum wire loop dipped into the original solution gave a yellowish-green tint to the Bunsen flame: *Presence of Ba*.

Found Ba, SO_4.

SECTION VI.

FULL ANALYTICAL COURSE AND TABLES.

After having tried the reactions of the analytical groups and learnt for each group how to detect its members occurring singly or two or more together, solutions containing a single member belonging to any of the groups, or members of two or more of these groups mixed, will readily be analysed.

370. *It is necessary first to separate the metals present into groups* by adding the group-reagents successively in such an order that each precipitates its own group only, leaving in solution the members of all other groups which may be present. This is effected by adding the group-reagents in the order directed in the General Table (419); refer to this and to the Group Table on pp. 168, 169.

HCl, which is the first reagent added, precipitates only Group I. leaving Groups II., III., IV., and V. in solution; hence if a precipitate is obtained on adding excess of HCl, after filtering it off any member or members of Group I. present will be obtained upon the filter, whilst the members of all other groups remain in the filtrate. On passing H_2S into the heated filtrate*, members of Group II. alone will be precipitated and may be filtered off; the filtrate*, after removal of H_2S and oxidation of any Fe present by boiling with HNO_3, may be mixed with AmCl and excess of AmHO; the AmHO will precipitate any members of Group III.A, Group III.B and Mg remaining dissolved by the AmCl; the addition of Am_2S to the filtrate* will separate Group III.B as sulphides. Group IV. is precipitated from the filtrate* by the last group reagent Am_2CO_3, and the filtrate* is examined for Group V., the members of which not being precipitated by any group reagent will now remain in solution if they were originally present.

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* Or solution, supposing no precipitate to have been obtained, and filtration therefore to have been unnecessary.

Since each group must be *entirely* precipitated by its own group reagent it is necessary, if any precipitate is formed, to add each group reagent in "excess" before proceeding to add the next: unless the presence of the reagent in excess is ascertained before proceeding, much confusion may be caused.

371 *Addition of reagents in excess.*—The most general method for ascertaining the presence of a reagent in excess is to add a few drops more of the reagent to the clear liquid obtained either by letting the precipitate settle or by filtering off a small quantity of it; if any further precipitate is formed the filtered portion must be returned, more of the reagent must be added, and the clear liquid again tried with a few additional drops of the reagent, this process being repeated until no further precipitate is caused.

In certain cases this method of proceeding is rendered unnecessary, the excess of a reagent being detectable by its smell after thoroughly mixing up the solution and blowing out the air above it; this is the case for example with H_2S and AmHO. In other cases the excess is seen by the colour of the filtrate, as with yellow Am_2S.

372. *Evaporation before precipitating Group III.*—Since certain organic substances hinder or prevent the precipitation of the members of Group III.A. by AmHO, it is necessary, if the preliminary examination has shown the presence of organic matter, to destroy it by evaporation and ignition before proceeding to precipitate Group III.: evaporation to dryness and gentle ignition are also necessary to separate SiO_2, which if remaining in solution might be mistaken for Al_2Ho_6. But if it is known that neither organic substances nor SiO_2 are present, a considerable saving of time may be effected by boiling the H_2S filtrate until it no longer smells of H_2S. then continuing to boil for several minutes after adding a few drops of strong HNO_3; this process may replace the evaporation to dryness and ignition.

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* Or solution, supposing no precipitate to have been obtained, and filtration therefore to have been unnecessary.

373. *General remarks.*—The ordinary method of analysis drawn out in the General Table and in the Tables for examination of the group precipitates will present little difficulty if the directions therein given are strictly complied with. Table III.C. will perhaps be found rather more difficult, and should not be attempted until some proficiency has been obtained by practice on the other Group Tables. Table III.D., which must be employed under conditions fully explained in (460), should not be used until considerable proficiency has been attained.

374. It will be seen that the full course of analysis is arranged so as not only to prove what is present, but also that all else is absent: this, except in special cases, is the object in view.

The process of analysis is much hastened by using separate portions of the solution instead of carrying on the examination throughout with one part of the solution; the plan in the General Table is however preferable, since it gives at once a notion of the relative quantities of the different substances present.

For an example of how to write out the results see pars. (488–493); it will be seen that the form adopted is that of the Analytical Tables.

PRELIMINARY EXAMINATION OF SOLID SUBSTANCES.

375. Before proceeding to employ the full course of analysis for substances of complex composition, the student will do well to examine a few solid substances for metals and acid-radicles by the tables given in paragraphs (387–418)*. For the full account of how to try these tests for any particular substance the student must refer back to the preceding reactions in Section IV.

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* For example of substances to be analysed by the Preliminary Examinations see paragraph (538).

The results of these analyses are entered in the form of the Table as shown in the examples in paragraphs (488) and (489). *The entries must be made during the progress of the analysis.*

376. *Preliminary Examination for Metals.*—A little study of this Table (387 *et seq.*) will show that it contains three main tests marked Exps. I., II., and III.; these stand in the first column: in the second column are placed the results which may be noticed on trying these experiments; the student should glance through these so as to be forewarned of what he has to look for. The main classification of the results which may be observed is indicated by large-type letters, the subsidiary divisions are marked by small numbers and italics, and the individual results are simply placed in succession in a vertical column. Occasionally an observation is made which it is advisable to confirm by an additional experiment: this "confirmatory" test is then entered in the first column, just below the observation to be confirmed, and is marked "confirm?" to distinguish it from the main experiment, its results are placed beside it in the second column: the student must understand that any one or more of the results noted in the second column may be looked for.

A few salts and mixtures such as those mentioned in (538) should be subjected to a careful preliminary examination by the student, and the results obtained carefully entered.

377. *Preliminary Examination for Acid-radicles.*—This Table (410–418) exactly resembles in arrangement the Table for metals which precedes it, and no explanation is therefore necessary. The student should examine by it some solid substances (538) for acid-radicles only.

378. A few solid substances may now be tested for both metals and acid-radicles by the two Preliminary Tables.

DETECTION OF ACID-RADICLES AND METALS BY PRELIMINARY TESTS AND BY EXAMINATION OF THE SOLUTION.

379. After having become familiar with the preliminary examination of solid substances the student may analyse some easy substances containing only one or two metals and acid-radicles. The analytical course drawn out for the examination of complex substances may be at once employed for these more simple analyses, as the student is thus enabled to prove not only the presence of any particular metals or acid-radicles, but also the absence of all others.

He will by this means be gradually introduced to the analytical course, and after satisfactorily analysing some simple substances, may proceed to mixtures of gradually increasing difficulty or complexity.

At first no mixture should be attempted which will yield a precipitate containing a phosphate in Group III., and members of Groups III.A. and III.B. should not be present together in the same solution, also substances containing organic matter and SiO_2 should be avoided; the examination of the filtrate after passing H_2S is thus very much simplified.

The following is a description of the course to be pursued in making the complete analysis of a complex substance.

GENERAL COURSE OF ANALYSIS FOR SOLID AND LIQUID SUBSTANCES.

The substance given for analysis* will be either a liquid or a solid, since the analysis of gases forms a department which is best studied by itself.

Unless it is known that cyanogen is not present in the substance, it must be tested for in a small portion by the "Prussian blue" or AmCyS test (304 or 306); if found, refer to (482 *et seq.*) for the preparation of the solution and the method of analysis : if cyanogen is not present the substance is examined by (380, 381) or by (382 *et seq.*) according as it is liquid or solid.

Silicates may also be tested for by fusing some of the finely powdered substance in a bead of microcosmic salt. (293.)

THE SUBSTANCE IS A LIQUID.

380. Test it with both blue and red litmus-papers, one of the following results will be obtained :—

It is neutral (not changing either paper).	*It is acid* (turning *blue* litmus *red*).	*It is alkaline* (turning *red* litmus *blue*).
Absence of acids and alkalis and of salts with acid or alkaline reaction. The salts of Ag and Mg and certain salts of Am, Na, K, Ba, Sr, and Ca are the only neutral soluble salts.	Presence of an acid, or salt with acid reaction.	Presence of a hydrate of Am, Na, K, Ba, Sr, or Ca, or of a salt with alkaline reaction.

381. Evaporate a few drops of the liquid upon a piece of platinum foil or a piece of thin glass or porcelain†, smelling it occasionally :—

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* For examples of substances to be analysed see par. (539).

† A piece of the side of a broken flask will serve well.

No residue is left:—The liquid must consist of some volatile substance, probably of water, either pure or containing certain gases or volatile substances dissolved in it (*e.g.*, NH_3, HCl, Br, &c.) which are detected by their smell, by the action of the liquid on litmus-paper, or by special tests. If the liquid has no action on litmus, no smell, and leaves no residue on evaporation, and is tasteless, it is pure water.	*A residue is left:*—Examine the liquid by the further preliminary tests in (334) or evaporate a portion of the solution to dryness in a porcelain dish, avoiding heating the substance after it is dry; examine the residue by the preliminary tables for metals and acid-radicles (387–418). Examine the larger part of the solution for metals by the general table (419) paying attention to (420), and the rest of the solution for acid-radicles by (440 *et seq.*).

THE SUBSTANCE IS A SOLID.

382. If the substance is metallic in appearance refer to (467) for the method of analysis, or if it is non-metallic proceed as is directed below.

Finely powder some of the substance in a mortar, using an agate mortar if it is very hard. A part of this powder is first examined by the Preliminary Tables for metals and acid-radicles (387–418).

PROCESS OF SOLUTION.

383. A large part of the remainder is boiled in a flask or boiling-tube with distilled-water, if it dissolves entirely see (384): if the powder does not dissolve, allow the liquid to stand still until the undissolved portion has in great part settled, then decant through a filter: the filtrate is solution I.:—

<table>
<tr><td colspan="4"><i>Residue in flask:</i> boil with dilute HCl (386), allow any residue to settle, decant through the filter used above (386 <i>a</i>); repeat this process and finally boil the residue with strong HCl, let settle, decant through the filter:—</td></tr>
<tr><td rowspan="3"><i>Filtrate is</i>
Solution II.</td><td colspan="3"><i>Residue:</i> heat with dilute, then with strong HNO_3 (386 <i>b</i>) as was directed above for HCl; decant through the filter:—</td></tr>
<tr><td rowspan="2"><i>Filtrate is</i>
Solution III.</td><td colspan="2"><i>Residue</i> may be warmed for a short time with a mixture of strong HNO_3 (386 <i>b</i>) with three times as much HCl; if this does not dissolve it, dilute and decant the acid mixture through the filter:—</td></tr>
<tr><td><i>Filtrate is</i>
Solution IV.</td><td><i>Residue:</i> wash well with water, dry, and examine as directed (475 <i>et seq.</i>) for substances insoluble in water and acids.</td></tr>
</table>

384. If at any of the preceding stages the substance dissolves entirely, proceed at once to examine the solution by the General Table (419), paying attention to (420) if the substance is entirely soluble in water.

385. The Solutions I., II., III., IV. need rarely be examined separately; a general method is to add to I. some HNO_3 (see 420) and then some HCl to the clear liquid; add also to the HNO_3 solution (III.) some HCl. Any precipitate thus caused by HCl is examined by Table I., and any precipitate caused then on mixing the solutions I., II., III., IV. is examined by (475) as an insoluble substance. It is best to boil down the solutions III. and IV. considerably before mixing them with I. and II., in order to get rid of the HNO_3 as much as possible; they are then mixed with dilute HCl, and any precipitate thus caused is examined by Table I.

386. Any changes which occur on adding HCl should be noted down; notice especially whether any gases are given off. Many of these gases may be detected by their smell, but a closer examination of them may be neglected, as they are obtained in the preliminary examination for acid-radicles (411). If the gases come off from the aqueous solution on adding HCl, or on mixing the HCl and aqueous solutions, they must have been present combined with metals as salts soluble in water, probably salts of alkalis; if they are evolved on treating the residue insoluble in water with HCl, they were present as salts insoluble in water.

386 *a.* On cooling the hot HCl solution crystals often separate, these consist probably of $PbCl_2$, they may be filtered off and dissolved in a little boiling water; if on addition of K_2CrO_4 to this solution a yellow precipitate forms, it shows the *presence of Pb.*

386 *b.* HNO_3 seldom requires to be used in dissolving a substance, and when necessary should be employed only in small quantity, since it is liable to cause a precipitate of S from H_2S. (423).

Note.—If a complex solid mixture is given for analysis, and the only object is to obtain a solution as rapidly as possible, it may be at once boiled with aqua regia for a few minutes, then diluted and filtered: the residue is examined as a substance insoluble in water and acids (475 *et seq.*), and the filtrate is evaporated nearly to dryness : dilute HCl is added, and the solution examined by the General Table (419).

PRELIMINARY EXAMINATION FOR METALS.

387. The substance is required in the solid state for this examination; hence if a solution is being analysed, some of is must be evaporated to dryness and the dry residue employed (see 381).

Carefully note down the appearance and physical properties of the original substance: whether it is crystalline or amorphous*; its hardness†, colour‡, smell, and anything which strikes you on a careful examination aided by a pocket-lens.

The following three experiments (388, 397, 398), together with some confirmatory ones when necessary, are then to be tried on small quantities of the finely powdered substance, and the results obtained noted down.

For a fuller explanation of the form of the Table, see paragraph 376.

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* A body, if crystalline, consists of particles which have a definite and similar shape: the particles are amorphous if they show no similar and definite shape.

† The hardness of a substance may be roughly tested by trying to scratch it with the point of a penknife : if very easily scratched, it is soft ; if the knife will not scratch it, but simply marks it is a lead pencil would do paper, it is very hard (*e.g.* Quartz).

‡ The salts of the following metals are coloured:—Cu, *blue;* Fe‴, *brownish-yellow;* Fe″, *pale-green;* Ni, Cr, $CuCl_2$, *green;* Mn, *delicate-pink;* Co, *reddish-pink.* Chromates, *light-yellow;* dichromates, *orange-red;* Sb_2S_3, CuO, MnO_2, *black;* HgO, Pb_3O_4, *red;* PbO_2, *dark-brown;* PbO, *light-brown;* Cr_2O_3, *green.*

Experiment.	Observation.	Inference.
388. Exp. I.—Heat a small quantity of the substance in a small test-tube or piece of hard glass tubing drawn off at one end.	A. The substance does not change.	Absence of organic substances which blacken and give off a smell of burning; of volatile substances which sublime; of substances which are altered by ignition; and of water combined with or absorbed by the substance, which would be evolved as steam and form drops on the side of the tube.
	B. The substance changes. 1. *It changes colour:—*	
	Yellow, hot. *white*, cold.	ZnO.
	Yellow, brown ,, *yellow* ,,	PbO.
	Yellow-brown ,, *yellow* ,,	SnO_2 or Bi_2O_3.
	Dark-red or *black* ,, *red-brown* ,,	Fe_2O_3.
	The substance blackens— *a.* Emitting a smell of burning. The black carbon may be burnt off by strongly heating on platinum foil with the blowpipe flame.	Presence of organic matter.
	Smell of acetone.	An acetate.
	Smell of burning sugar.	A tartrate.
	b. Emitting no smell of burning. The colour is not removed by ignition as under *a.*	Probable presence of Co- or Cu-salts.
	2. *It fuses, and becomes solid again on cooling.*	Salts of alkalis or certain salts of the alkaline earths.
	3. *It sublimes.*	Compounds of Am, As, Hg; or free S or I.
Confirmatory.—The sublimate is carefully examined, if necessary with the aid of a lens.	White, crystalline (sparkling) sublimate,	$HgCl_2$, As_2O_3.
	White, non-crystalline sublimate.	Probably Am-salt.
	Black sublimate, becomes red when rubbed.	HgS.
	Yellow sublimate.	As_2S_3. HgI_2, becomes scarlet when rubbed.
	Sublimate of reddish drops, which are yellow when cold.	S free or from certain polysulphides.
	Violet vapour, cooling to blackish crystals.	Presence of I.
389. *Confirmatory.*—Heat another portion of the substance, mixed with about three times as much Na_2CO_3 in fine dry powder and a little KCy, in a tube closed at one end (147).	Grey mirror, which, when rubbed with a splinter of wood or a glass rod, or when examined by a lens, is seen to consist of globules of Hg.	Presence of $Hg^{\cdot}$.
	Blackish-brown shining mirror, and smell of garlic; no globules.	Presence of As.

Experiment.	Observation.	Inference.
390. *Confirmatory.*—Pour a little strong KHO solution upon some of the substance and heat to boiling; or mix some of the substance with soda-lime in a mortar, and moisten and heat.	NH_3 is given off, known by its smell and by turning moist red litmus-paper blue or turmeric-paper brown; the change in colour is only a trustworthy indication, if it occurs very soon, as the NH_3 in laboratory air often causes it to occur after a time.	Presence of NH_4.
	4. *The substance gives off water.*	Presence of water, absorbed or combined.
	It fuses first, then gives off water, and again becomes solid if the heat is continued.	Water of crystallisation.
	It swells up considerably whilst giving off its water.	Borax and certain borates, alums, and phosphates.
391. *Confirmatory.*—Examine the drops of water on the sides of the tube with blue and red litmus-papers.	The water is alkaline.	Probably NH_4-compounds.
	The water is acid.	Presence of volatile acids, *e.g.*, HNO_3, HCl, H_2SO_4.
	5. *Gas or vapour is given off.** *a. It is without smell.*	
392. *Confirmatory.* — Introduce a burning splinter of wood into the tube.	It burns more brightly, and if introduced with a spark at the end is inflamed,	O from chlorates, nitrates, peroxides, &c.; or possibly N_2O.
	The flame is extinguished.	CO_2 or N.
393. *Confirmatory.* — Introduce a glass rod, moistened with lime-water, into the tube.	The lime-water turns milky.	CO_2 from carbonates, oxalates, &c.
	The lime-water does not turn milky, but the flame was extinguished (392).	N, from $AmNO_2$ probably.
	b. The gas or vapour has a smell.	
394. *Confirmatory.* — Introduce a glass rod or slip of paper moistened with $K_2Cr_2O_7$.	Smell of burning S. The $K_2Cr_2O_7$ turns green; moist blue litmus is also reddened by the gas.	SO_2 from combustion of free S, or from acid sulphites, hyposulphites, reduction of sulphates or oxidation of sulphides.
	Reddish-brown nitrous fumes are given off, known by their peculiar smell, and by not colouring starch paste orange-red.	Nitrates of heavy metals (*e.g.* of Pb, Bi, Hg. &c.)
	Smell resembling Cl:—	
395. *Confirmatory.* — Introduce a glass rod carrying a piece of moist litmus.	Yellowish gas, which bleaches moist litmus.	Cl from certain chlorides.
396. *Confirmatory.* — Introduce a glass rod with moist starch powder on its end.	Brown vapour, which colours the moist starch powder orange-red.	Br from certain bromides.
	Violet vapour, which colours starch solution blue.	I, free or from certain iodides.

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* This will usually be shown by effervescence of the substance; if the gas or vapour is not detected by its colour or smell, it can only be found on testing for it specially by (392) and (393).

Experiment.	Observation.	Inference.
	c. The gas can be ignited, and burns at the mouth of the tube with a:—	
	Pale *yellowish-green* flame, more or less explosive.	NH_3 probably from strongly heated NH_4NO_3.
	Bright *white* flame, producing white fumes; the unburnt gas smells of garlic.	PH_3 probably from a hypophosphite.
	Peach-blossom coloured flame.	Cy from a cyanide, probably from $HgCy_2$.
397. Exp. II.—Dip a moistened loop of platinum wire, which gives no colour to the Bunsen flame, into the substance, and hold the loop with the adhering powder near the top of the Bunsen flame, or in the inner blowpipe flame. Then moisten with a drop of strong HCl and heat again. Continue the heating until no further change occurs in the colour of the flame.	A. The substance colours the flame:	
	1. *Intense yellow.*	Na.
	Examine the flame through the indigo-prism; a crimson colour is seen.	K or Sr, or both, are present.
	2. *Pale violet,* crimson through the indigo-prism.	K.
	3. *Yellowish-green.*	Ba probably.
	4. *Crimson,* same through the indigo-prism.	Sr.
Note. If silicic acid is present, the colorations for K and Na usually show only after ignition of the powdered substance with powdered $CaSO_4$.	5. *Orange-red,* dingy-green through the indigo-prism.	Ca.
	6. *Bright green.*	Cu or B_2O_3.
	A blue colour is seen after moistening with strong HCl.	Cu.
	7. *Blue* { *Intense.*	$CuCl_2$, $CuBr_2$.
	Livid.	As, Sb, Pb.
	B. The substance does not colour the flame.	Probable absence of the above.
398. Exp. III.—Heat the substance in a small cavity scooped in a piece of wood-charcoal, in the blowpipe flame.	A. The substance decrepitates, or crackles.	NaCl and certain other crystalline salts.
	B. The substance deflagrates, or causes charcoal to burn rapidly.	Chlorates, nitrates, &c.
	C. The substance fuses easily, and is absorbed by the charcoal, or forms a liquid bead.	Salts of alkalis and certain salts of the alkaline earths.
	D. An infusible residue is left on the charcoal.	
	1. *The residue is white and very luminous.*	Probably BaO, SrO, CaO, MgO, Al_2O_3, ZnO (or SiO_2).
399. *Confirmatory.*—Detach a portion of the cool residue from the charcoal, place it upon a piece of red litmus-paper, and moisten with a drop of water.	The paper turns blue, showing the residue to be alkaline.	BaO, SrO, CaO, and possibly MgO.

Experiment.	Observation.		Inference.
400. *Confirmatory.*—Moisten the residue on the charcoal when cool with several drops of $Co(NO_3)_2$ solution, and heat again strongly in the outer blowpipe flame.	A *blue* residue, the colour of which does not disappear with intense heat.		Al_2O_3, and some phosphates, silicates, and borates.
	A *pink* residue.		MgO.
	A *green* residue.		ZnO.
401. *Confirmatory.*—Heat a small quantity of the substance in a clear colourless borax bead, first in the outer, then in the inner blowpipe flame.	2. *The residue is coloured.*		
	Colour of bead:		
	In outer flame.	In inner flame.	
	Green, hot. *Blue*, cold.	*Colourless*, or *red*, cold.	Cu.
	Blue, hot and cold.	*Blue*, hot and cold.	Co.
	Violet, hot. *Yellow*, cold.	*Grey* or *opaque*.	Ni.
	Brown-red, hot. *Yellow*, cold.	*Green*, hot and cold.	Fe.
	Green, hot and cold.	*Green*, hot and cold.	Cr. See (402).
	Reddish-purple, hot and cold.	*Colourless*, hot and cold.	Mn.
402. *Confirmatory.*—The presence of Mn and Cr, may be confirmed by fusing the substance with Na_2CO_3 and KNO_3 on platinum foil.	*Light-yellow* mass on cooling.		Cr.
	Blue-green mass on cooling.		Mn. (See 406.)
	3. *The residue is coloured, or metallic scales or globules are seen.*		
403. *Confirmatory.*—Heat also some of the substance, mixed or covered with powdered KCy and Na_2CO_3 or $K_2C_2O_4$, on charcoal in the inner blowpipe flame.	The substance is reduced to the metallic state. (See 407.)		Au, Ag, Cu, Pb, Sn, Sb, Bi; [Co, Ni, Fe, Mn, form grey powders].
	a. Without incrustation.		Au, Ag, Cu, Co, Ni, Fe, Mn.
	Brilliant white metal.		Ag.
	Yellow metal.		Au.
	Red scales or globules.		Cu.
	Grey powder, attracted by a magnetised knife-blade (407).		Fe, Co, Ni, Mn.
	b. With incrustation.		
	Globules.	Incrustation.	
404. *Confirmatory.*—Detach several of the globules with the point of a knife, and strike them on the bottom of an inverted mortar with the pestle; if they flatten to a cake they are *malleable*, if crushed to a powder they are *brittle*. Try also if a globule, fixed on the point of a knife, marks paper.	*White, malleable,* not marking paper.	None, or very slight.	Sn.
	White, brittle.	*Orange*, hot. *Yellow*, cold.	Bi.
	White, malleable, marking paper.	*Yellow*, hot and cold.	Pb.
	White, brittle, giving white fumes.	*White*, close to substance.	Sb.
Note.—The appearance and malleability of one reduced metal is frequently much altered by the presence of another.	*White* or none.	*Yellow*, hot. *White*, cold.	Zn.
	White or none.	*Red-brown*, easily volatilised.	Cd.
	None.	*White*, and white fumes; smell of garlic.	As.

Experiment.	Observation.	Inference.
405. Exp. IV. — Fuse some of the substance, mixed with Na_2CO_3, on charcoal in the inner blowpipe flame *produced from a spirit-lamp;* remove the fused mass when cold, place it on a bright silver coin, moisten with a drop of water, and let stand for several minutes.	On rinsing off the substance, the coin is found to be stained black (408). Also, if a drop of HCl be placed upon the mass, a smell of H_2S is perceived, and lead-paper is blackened.	Presence of S, free or combined. *Note.* — Since this reaction serves to detect S and sulphur-acid-radicals, it more properly belongs to the Preliminary Examination for acid-radicles: it is, however, best tried here.

NOTES TO THE PRECEDING PRELIMINARY TABLE.

406. The green colour produced by Mn conceals the light yellow ue to Cr; on boiling the residue with water it gives a pink or purple solution, the colour of which is best seen on filtering: this colour is due to the formation of $KMnO_4$, and shows Mn to be present. On acidifying this solution with $H\bar{A}$ and boiling again for several minutes, the purple colour is destroyed; and on filtering, the yellow colour due to Cr is seen: the presence of Cr may be further confirmed by the formation of a yellow precipitate in the acid solution on addition of $Pb\bar{A}_2$.

407. The metal is best separated and examined by detaching the mass when cold from the charcoal, and powdering it by crushing in a mortar or on a watch glass with a little water, letting stand for a short time, and then quickly pouring off the water down a glass rod or pestle which is wetted and pressed against its edge; by several times repeating this operation, the heavier metallic particles alone are left in the mortar or watch glass (33 *a*).

408. This stain is removed by rubbing the coin with a little lime made into a paste with water.

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409. *Note.*—When the substance to be examined contains several bodies, they frequently more or less mask one another's reactions: thus Co if mixed with Fe will give a bead green whilst hot and blue when cold, thus resembling Cu, but differing in remaining blue in the inner flame: hence the composition of many complex mixtures is only roughly indicated by the Preliminary Examination, and must be confirmed and established in the wet way. Many substances, more particularly minerals, however, can be completely analysed by a careful Preliminary Examination.

PRELIMINARY EXAMINATION FOR ACID-RADICLES.

410. The substance is required for this examination in the state of powder or of *strong* solution.

If the substance given for analysis is a solution, part of it may be evaporated to dryness: the dry residue is then finely powdered and subjected to the following tests. If several acid-radicles and metals are present, the reactions may be more or less perfectly concealed or altered, hence failure in obtaining a certain reaction does not in all cases necessarily prove the absence of an acid-radicle.

Experiment.	Observation.	Inference.
411. Exp. I.—Treat some of the substance with dilute HCl and note the result, then heat moderately (416).	*One or more of the following gases may be evolved:—*	
	A colourless gas without smell, which turns milky a drop of lime-water on the end of a glass rod.	CO_2 from a carbonate.
	A gas of suffocating smell which turns a drop of $K_2Cr_2O_7$ solution green.	SO_2 from a sulphite.
	Yellow S is precipitated at the same time.	SO_2 and S from a theiosulphate*.
	A gas with fetid odour, which blackens a drop of $Pb\bar{A}_2$ solution, or a piece of lead-paper (417).	H_2S from a sulphide; or possibly from a sulphite or hyposulphite, if Zn or other reducing agent be present.
	Reddish fumes.	N_2O_3 from a nitrite.
	A yellowish-green gas, of suffocating smell, and which bleaches moist litmus-paper, is evolved:—	
	a. In the cold; and also evolved on adding $H\bar{A}$.	Cl from hypochlorites.
	b. Only when heated.	Cl from action of HCl on oxidising substances, *e.g.*, MnO_2, chromates, nitrates, chlorates, &c.
	A colourless gas, smelling of bitter almonds.	HCy from a cyanide.

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* Formerly called a "hyposulphite."

Experiment.	Observation.	Inference.
412. Exp. II. — Heat another portion gently with strong H_2SO_4. *Note.* — No attention need be paid to gases evolved in Exp. I. (411).	1. A gas is evolved with pungent smell, which fumes in the air, and renders milky a drop of $AgNO_3$ solution made acid with HNO_3.	HCl, HBr, HI, HF* from chloride, bromide, iodide, or fluoride.
Confirmatory.—Mix some of the substance with MnO_2 free from chloride, add strong H_2SO_4 and warm.	A yellow gas is given off with strong smell, and which bleaches moist litmus-paper (418).	Probably Cl from a chloride.
	Brown vapour is given off, which colours a little moist starch powder orange-red.	Br from a bromide.
	Violet vapour is given off, which colours a drop of starch-paste blue.	I from an iodide.
	2. A heavy fuming suffocating gas is evolved, and the liquid behaves as if the glass were greasy; on rinsing out the tube, and drying it thoroughly, the inside is seen to be corroded and dimmed.	HF from a fluoride. *Note.*—The HF acting upon the silica of the glass evolves SiF_4, which is detected by holding a moistened glass rod in the gas, SiO_2 is deposited as a gelatinous film upon it.
413. *Confirmatory.*—Drop into the hot liquid a few small pieces of copper.	3. The acid fumes are reddish, usually seen only on *heating* after adding a few fragments of Cu.	Presence of a nitrate. *Note.*—If iodide is present it must usually be separated by Cu_2SO_4 (266) before a nitrate can be detected.
414. *Confirmatory.*—Boil some of the substance with water, or take some of the solution if a liquid; cool, add about twice as much strong H_2SO_4, cool, and pour cold solution of $FeSO_4$ carefully in upon the top of the acid liquid.	A dark brown ring or layer forms upon the surface of the acid, best obtained on cooling the liquid by immersing the test-tube in cold water.	Presence of a nitrate.
	4. Greenish-yellow gas, smelling like Cl, and exploding when warmed. Confirm by adding indigo solution and H_2SO_3 to the colourless aqueous solution of the substance (252). For detection of a nitrate and chlorate when mixed, see (256).	Presence of a chlorate.
	5. The substance changes from yellow to green, O being evolved as is shown by a glowing taper.	Presence of a chromate.

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* HF would also render the drop milky by depositing SiO_2 from SiF_4, formed by its action on the glass, but it is distinguished by corroding the glass. (See 412, 2, note.)

Experiment.	Observation.	Inference.
	6. Yellowish-green gas, with suffocating smell, which bleaches moist litmus.	Cl from a hypochlorite (detected already in 411), or from a chloride in presence of MnO_2 &c.
	7. The same gas as in 6, but coloured reddish-brown by nitrous fumes:	A chloride in presence of a nitrate or nitrite.
	8. Or coloured reddish-brown by $CrOCl_2$.	A chloride in presence of a chromate.
	9. Substance does not blacken, but evolves CO which burns with a blue flame, and CO_2 which turns a drop of lime-water milky.	Presence of an oxalate.
	10. Substance blackens, and SO_2 is smelt.	Presence of a tartrate.
	11. A smell of acetic acid or vinegar is noticed.	$H\bar{A}$ from an acetate.
415. *Confirmatory.*—Add a little alcohol to the substance and heat with H_2SO_4.	Strong fragrant odour.	Ethyl-acetate from an acetate.

NOTES ON THE PRECEDING TABLE.

416. If HCl has been employed in dissolving the substance, the acid-radicles detected by the action of HCl will have been already noticed in preparing the solution for metals (383).

417. The S present in sulphides which are not decomposed by dilute HCl, is detected by fusing the powdered substance with two or three times as much fusion mixture ($Na_2CO_3 + K_2CO_3$) in a covered crucible, boiling with a little water, and placing a drop upon a bright silver coin, when a dark stain will be produced; or by adding excess of an acid to the solution, when H_2S will be evolved. Smaller quantities of S may be detected by the formation of a black colour or precipitate on adding $Pb\bar{A}_2$ to the solution.

418. Chlorides treated in this manner evolve Cl, recognised by its colour, smell, and bleaching action on litmus-paper; but this test for chlorides is reliable only if the precautions stated in (259) are attended to, since many samples of MnO_2 evolve Cl when warmed alone with H_2SO_4.

EXAMINATION FOR METALS.

After the preceding Preliminary Examinations have been made, a solution of the substance is made as directed in (383 *et seq.*), and this solution is further examined in a more systematic manner. This is often called "The Examination in the Wet way." See next page.

419. GENERAL TABLE FOR SEPARATION OF

If the substance for analysis is a liquid with neutral or *alkaline* reaction, it
examined as directed below.

In this Table, and also in the Group Tables which follow, it has been sup-
precipitate will be produced when the reagent is added for its detection ; and

<table>
<tr><td colspan="3">Add to the greater part of the "original solution"* HCl; if any precipitate is produced,</td></tr>
<tr><td rowspan="3">The precipitate may contain—
$PbCl_2$—<i>white.</i>
$AgCl$—<i>white.</i>
Hg_2Cl_2—<i>white.</i>
(See 422.)
Examine the precipitate by Table I. (433).
<i>Note.</i>—The absence of Pb is not proved, if it is not precipitated in this group, as $PbCl_2$ is somewhat soluble, and Pb may therefore be found only in Group II.</td><td colspan="2">The filtrate or solution, which must give no further precipi-
then diluted with water (425), unless it is already dilute;
warmed gently (426, 426 a) and filtered :</td></tr>
<tr><td rowspan="2">The precipitate may contain—
HgS—<i>black</i>
PbS—<i>black</i>
Bi_2S_3—<i>black</i>
CuS—<i>black</i>
CdS—<i>yellow</i>
} Insoluble in KHO and in Am_2S.
SnS—<i>brown</i>
SnS_2—<i>yellow</i>
Sb_2S_3—<i>orange</i>
As_2S_3—<i>yellow</i>
[Au_2S_3—<i>black</i>]
[PtS_2—<i>black</i>]
} Soluble in KHO and in Am_2S.
Examine the precipitate by Table II. (435).
<i>Note</i> 1.—This precipitate should not be examined at once, as possibly a precipitate may be formed on boiling the filtrate, and this would have to be added to the above.
<i>Note</i> 2.—Unless the liquid has been sufficiently diluted some of the above metals may be afterwards precipitated by Am_2S in Group III., and cause much confusion.</td><td>The solution or filtrate is di-
cipitated: as soon as H_2S
dish until it ceases to smell
and the residue gently ignited
heated, then water is added;
Add a few drops of the acid
tate shows the <i>presence of a</i>
Add to the rest of the HCl solu-
once any precipitate which
(429): if a phosphate is
phates are absent proceed as
[<i>Note.</i>—Refer to</td></tr>
<tr><td>The precipitate may contain—
Al_2Ho_6—<i>almost colourless.</i>
Cr_2Ho_6—<i>light-green.</i>
Fe_2Ho_6—<i>reddish-brown.</i>
Examine the precipitate by Table III. A (436).</td></tr>
</table>

NOTES TO THE GENERAL TABLE.

420. If the liquid under examination is alkaline or neutral in reaction, before commencing the examination by the General Table, HNO_3 must be added in slight excess ; if no precipitate forms, proceed at once to examine the liquid by the General Table ; if a precipitate is produced, more HNO_3 is to be added, and the liquid heated ; should

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* By the "original solution" is meant the solution of the substance if a solid, or the substance itself if a liquid, to which no reagent has been added.

METALS INTO GROUPS BY GROUP-REAGENTS.

requires special treatment (420); if its reaction is *acid*, it may be at once

posed that all metals are present: if a metal or group of metals is absent, no
hence no filtration is necessary, the solution being then treated as a filtrate.

---

continue to add HCl as long as it causes any further precipitate (421), warm and filter:

---

tate on addition of several drops of HCl, is boiled down if necessary (423), and is
it is then saturated with H_2S by passing the gas through the solution * (500 *a*), and

---

luted and H_2S is passed again into it, to make sure that Group II. is completely pre-
produces no further precipitate the clear solution or filtrate is boiled in a porcelain
of H_2S (427), a little strong HNO_3 is then added, and the liquid is evaporated to dryness
in the dish. When the dish is cool, a little strong HCl is poured upon the residue and
any undissolved residue is filtered off (428).

solution to some $AmHMoO_4$ solution in a test-tube, and warm gently; a yellow precipi-
phosphate; if no precipitate is formed with $AmHMoO_4$, *phosphates are absent.*

tion AmCl, heat it to boiling, then add AmHO in excess, boil again and filter off at
may form as quickly as possible, keeping the funnel closely covered with a glass plate
present and a precipitate is produced by AmHO refer to Table III.D. (466), if phos-
is directed below:—

(463, *et seq.*), for full directions for precipitating Groups III.A and III.B.]

Add to the filtrate or solution, which may be coloured (430), Am_2S in excess, boil and filter:—

The precipitate may contain— ZnS—*white.* MnS—*light-pink.* CoS—*black.* NiS—*black.* Examine this precipitate *at once* by Table III.B (437).	Add to the filtrate or solution, which must be distinctly yellow or brown (431), Am_2CO_3, warm gently and filter:—	
	The precipitate (432) may contain— $BaCO_3$—*white.* $SrCO_3$—*white.* $CaCO_3$—*white.* Examine the precipitate by Table IV. (438).	The filtrate may contain Mg, K, Na. Examine by Table V. (439).

a precipitate still remain, it is filtered off and the filtrate examined by the General Table, commencing with the addition of HCl.

The precipitate caused and not dissolved by HNO_3 may be finely divided sulphur, separated as a *white* powder from a sulphide, or *yellow* from a theiosulphate †; this precipitate is recognised by its colour, and by not being separable by standing or filtration, it may be disregarded; there may also be precipitated H_4SiO_4 (gelatinous, almost colourless), SnO_2 or metastannic acid (see 420 *a*), Sb_2O_5, AgCl (all three white), SnS_2, As_2S_3 (both yellow), Sb_2S_3 (orange). Hence a full examination of this precipitate for all that it may contain will involve boiling the precipitate with aqua regia, diluting and filtering; the insoluble residue is examined as a substance insoluble in water and acids (475) and the aqua regia solution is added to the filtrate from the precipitate caused by HNO_3, any precipitate caused on thus mixing the solutions being examined by Table I. (433).

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* Instead of diluting and passing the gas, H_2S-solution may be added.

† Often called a "hyposulphite."

420 *a*. Metastannic acid is precipitated from a solution of an alkaline-metastannate by addition of an acid: it forms a white precipitate which becomes *golden-yellow* when moistened with $SnCl_2$ solution; it is further recognised by its dissolving in the washing-water whilst being washed, but being reprecipitated when the washings run into the acid filtrate.

421. Even if HCl has been used in dissolving the substance, it is advisable to add a few drops of the acid to make sure that no member of Group I. remains.

422. It must be remembered that HCl also precipitates strong Ba solutions; this precipitate however disappears as $PbCl_2$ does on diluting with water and heating, thus differing from AgCl and Hg_2Cl_2.

423. If HNO_3 or aqua regia has been used in dissolving the substance, or if the solution smells of Cl or SO_2, it should be boiled down considerably until after adding HCl and boiling, neither of the above gases is smelt; it is then diluted, and H_2S passed at once whether the dilution has caused any precipitate or not.

If As has been detected in the preliminary examination, H_2SO_3 should be added until the liquid smells of SO_2 after having been boiled for a short time, and the liquid is then heated for some time short of boiling until it no longer smells of SO_2. This treatment reduces arsen*ic* and stann*ic* compounds to the arseni*ous* and stann*ous* condition. In the case of As this reduction is to be recommended, since arsen*ic* compounds are not easily precipitated by H_2S; it is also very desirable to reduce stannic compounds, since SnS_2 unlike SnS readily runs through the filter, and being yellow in colour may be mistaken for S. It will of course be necessary to ascertain by special tests (187–190, and 201–203), made on the *original solution*, in which state As and Sn were originally present. If any precipitate is produced by boiling with H_2SO_3 other than white sulphur, refer to (424).

424. Whilst boiling with H_2SO_3 some H_2SO_4 is usually formed, which may partially or completely precipitate Pb, Ba, Sr as white sulphates; H_2SO_3 will also possibly precipitate Au, which causes the cool liquid to appear blue by transmitted and red by reflected light, and on boiling separates as a black powder. Examine any precipitate formed by H_2SO_3 by Table G (434).

425. On addition of water, Bi, Sb, Sn may give white precipitates of their oxychlorides; these precipitates may be disregarded, since H_2S readily converts them into sulphides.

426. H_2S often causes a fine *white* precipitate of S, owing to the presence of certain oxidising substances, such as Cl, HNO_3, $HClO_3$, H_2CrO_4, H_2MnO_4, $HMnO_4$, H_2SO_3, Fe_2Cl_6, &c.; this precipitate is known by its perfect whiteness, and by not being separable by standing or filtration, it may be neglected; great care must however be taken not to mistake SnS_2 for S, it resembles S in running through filter-paper, but is distinctly *yellow* in colour, it may often be coagulated by being shaken or heated.

426 *a.* Certain changes may be observed whilst H_2S is being passed, they should be carefully noted. Thus Pb and Hg during precipitation by H_2S show characteristic colours (149, 144); a solution coloured reddish-yellow by H_2CrO_4 becomes green; and a solution coloured green by H_2MnO_4 or purple by $HMnO_4$ becomes colourless.

427. If on boiling the filtrate or solution after passing H_2S a yellow precipitate forms, this shows the presence of an arsen*ic* or a stann*ic* compound; in this case pass H_2S into the boiling liquid as long as it causes any further precipitate, filter off and add this precipitate to any precipitate already obtained by H_2S to be examined by Table II.

428. SiO_2 and other substances may be present in this residue, but SiO_2 is distinguished from them all by readily dissolving in hot KHO solution.

The other substances which may remain undissolved by HCl are Al_2O_3, Fe_2O_3, Cr_2O_3, rendered difficultly soluble by the ignition to which the residue has been subjected; they are dissolved by long-continued heating with strong HCl, and the solution is added to the other solution which is to be examined for Group III.A.

$BaSO_4$ and $SrSO_4$ may also remain undissolved; they are detected by heating some of the residue strongly for a short time in the inner blowpipe flame upon a loop of platinum wire, then moistening the loop with a drop of strong HCl and holding it in the outer part of the Bunsen flame; crimson-red flashes, appearing red through the indigo-prism prove the presence of Sr, a yellowish-green flame coloration shows Ba.

A less ready method of detecting Ba and Sr is to dry the insoluble residue, fuse it on platinum foil with three or four times as much fusion mixture; allow it to cool, then boil with water until the mass is disintegrated, filter, wash the residue well upon the filter, and examine it by Table IV. (438) for Ba and Sr only.

429. Mn and Zn are very liable to be precipitated with Fe_2Ho_6, Al_2Ho_6, Cr_2Ho_6. The precipitation of Mn is partly prevented by keeping the liquid from exposure to the air after adding AmHO: but the further precaution should be taken of dissolving the precipitate produced by AmHO in HCl and reprecipitating it by AmHO, then filtering and adding the filtrate to the original filtrate from the Group III.A precipitate: this treatment of the precipitate should be repeated several times, if small quantities of Zn or Mn have to be looked for in presence of Al or Fe; or better still Groups III.A and III.B may be precipitated together (459) and the precipitate examined by Tables III.C (464, 465). See rules (463).

430. The filtrate or solution, after adding AmCl and AmHO, may be coloured *blue* by Ni, or *reddish-violet* by Cr_2Ho_6 which is dissolved in the excess of AmHO.

The Cr_2Ho_6 must be removed before proceeding to examine for the three remaining Groups; it is readily separated as a pale-green flocculent precipitate by boiling the liquid for a short time in a porcelain dish, adding a little more AmHO if the liquid does not smell of NH_3 after being boiled for a short time; this precipitate is filtered off, and the filtrate, which is colourless if the Cr has been entirely removed by boiling the liquid sufficiently long, is further examined for Groups III.B, IV., and V. by the General Table.

The coloration due to Ni differs from that caused by Cr_2Ho_6 in not being thus removed by boiling the liquid.

431. A *brown* or *dark coloured* filtrate shows that Ni is present*, some of the NiS precipitated by Am_2S having been dissolved in the excess of Am_2S and giving the filtrate its dark colour. If this coloration is noticed the filtrate should be poured into a porcelain dish and boiled, occasionally adding water if necessary, until on removing the lamp for a short time the black NiS settles, leaving a colourless liquid. The liquid is then run through a small filter and the black precipitate tested for Ni by fusing a portion of the filter-paper stained by the NiS in a colourless borax-bead: if a bead is obtained, which when heated in the outer flame is *violet* or *brown* whilst hot and *yellow* when cold, and *which becomes grey or opaque in the inner flame*, Ni is certainly present and need not be further tested for in Table III.B.

The colourless filtrate is examined by the General Table for Groups IV. and V.

432. Part of the precipitate produced by Am_2CO_3 often adheres firmly to the inside of the vessel in which it has been produced, in this case rinse the tube out several times with distilled water and then dissolve the precipitate by pouring in some hot $H\bar{A}$, causing it to run over the inside of the tube, and add this solution to the $H\bar{A}$ solution of the Am_2CO_3 precipitate made in Table IV.

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* If Groups III.A and III.B have been precipitated together by AmCl, AmHO, and Am_2S added in succession (459) the brown coloration of the filtrate may also be due to Cr_2Ho_6: on boiling the filtrate coloured by Cr_2Ho_6 in a porcelain dish for several minutes, *pale green* Cr_2Ho_6 is precipitated, which cannot be mistaken for *black* NiS.

433. TABLE I.—SILVER GROUP.

The precipitate produced by HCl may consist of $PbCl_2$, AgCl, and Hg'_2Cl_2: the precipitate on the filter is washed five or six times with boiling water, the first portions of the washings being kept apart :—

<table>
<tr><td rowspan="2">The first portions of the washings may contain $PbCl_2$ in solution, which if present in any quantity will be deposited in brilliant crystals on cooling.
Test for, or confirm the presence of, Pb, by adding to the washings K_2CrO_4: a yellow precipitate readily soluble in KHO shows :—
Presence of Pb.</td><td colspan="2">*Residue:* pour upon the precipitate on the filter some hot AmHO; when this has run through, heat it again, and pour it once more upon the filter :—</td></tr>
<tr><td>*Filtrate:* add HNO_3 until the liquid is acid: a white precipitate uniting into curdy particles when shaken or boiled shows :—
Presence of Ag.</td><td>*Residue* on the filter is black: dry at a gentle heat, best in a steam-bath; scrape the precipitate off the filter, mix with some dry Na_2CO_3, and heat in a small ignition tube (147). Globules of Hg form on the sides of the tube, often appearing only when the inside of the tube is rubbed or examined with a lens:—
Presence of Hg' (Mercurosum).</td></tr>
</table>

434. TABLE G.—EXAMINATION OF THE PRECIPITATE FORMED ON BOILING THE HCl SOLUTION WITH H_2SO_3. (See par. 424.)

The precipitate produced by H_2SO_3 may contain Au, $PbSO_4$, $BaSO_4$, and $SrSO_4$; the last three substances are perfectly white, and Au, if present, is therefore usually seen by colouring the precipitate brown or black; the Au is also usually visible during precipitation by H_2SO_3, or after the white sulphates have subsided, since it colours the liquid blue by transmitted and reddish by reflected light.

Ba and Sr can often be at once detected, if present, by taking a little of the moist precipitate on a loop of platinum wire, heating in the inner blowpipe flame for some time, moistening with HCl, and examining the flame coloration; but a more trustworthy method of proceeding is the following :—

Rinse the precipitate into a small porcelain dish, using as little water as possible; dissolve in the liquid a small crystal of $H_2\bar{T}$ or add a few drops of strong $H\bar{A}$; then add AmHO until it is just in excess, and boil for a short time, let stand and decant through a filter :—

<table>
<tr><td rowspan="2">*Solution* may contain $PbSO_4$; add $H\bar{A}$ in excess, then K_2CrO_4; a yellow precipitate soluble in KHO shows :—
Presence of Pb.</td><td colspan="2">*Residue:* pour upon the residue in the dish a little HCl and a few drops of HNO_3, heat to boiling, let stand, decant into a porcelain dish :—</td></tr>
<tr><td>*Solution:* boil until the smell of Cl is removed; add several drops of fresh $FeSO_4$ solution; a blue coloration and reddish precipitate show:—
Presence of Au.</td><td>*Residue:* dry by gently heating the dish, fuse with fusion mixture on platinum foil, and examine the mass for Ba and Sr as directed in the last part of (428).</td></tr>
</table>

435. TABLE II.—COPPER AND

The precipitate produced by H_2S in the HCl solution, after having washing water, collected in a test-tube, give no precipitate or only the filter and boiled with KHO or a small quantity of Am_2S*, and

GROUP II.A.—COPPER GROUP.

<table>
<tr><td colspan="5">**435.** *a. The residue* may contain HgS, PbS, Bi_2S_3, CuS, CdS. After having washed the precipitate and allowed it to drain, remove it from the filter into a porcelain dish (26 *d*), pour upon it strong HNO_3 sufficient to cover it completely, and heat gently as long as any red fumes come off, adding more strong HNO_3 if necessary to prevent evaporation to dryness. Boil off nearly all the acid, add a little water and dilute H_2SO_4, let stand for some time, stirring occasionally, and filter :—</td></tr>
<tr><td colspan="2">*Residue* may contain HgS (*black*), $PbSO_4$ (*white*), see note 1.
Remove the precipitate into a porcelain dish, using as little water as possible; add a little $H\bar{A}$, then AmHO in excess, boil and filter :—</td><td colspan="3">*Filtrate* may contain Bi, Cu, Cd.
Add AmHO in excess, boil and filter :—
[*Note.*—A blue coloration shows presence of Cu.]</td></tr>
<tr><td rowspan="2">*Residue:* dry on the filter at a gentle heat, best in the steam oven. Scrape the dried residue off the filter (note 2), and heat it, mixed with dry Na_2CO_3, in a small ignition-tube; globules of Hg form on the sides of the tube, which become visible when rubbed or when looked for by a lens :—
Presence of Hg'' (Mercuricum).</td><td rowspan="2">*Filtrate:* Add $H\bar{A}$ in excess, then K_2CrO_4, a yellow precipitate :—
Presence of Pb.</td><td rowspan="2">*Precipitate* (not easily seen in dark blue solutions): wash with hot water, dissolve off the filter by pouring upon it *a few drops* of boiling dilute HCl, pour this solution into a large quantity of cold distilled water, a milkiness appearing at once or after stirring and letting stand for some time, shows :—
Presence of Bi.
Note.—A precipitate produced by AmHO must always be tested as above for Bi, since Pb and Hg might possibly appear here as white precipitates and be mistaken for Bi.</td><td colspan="2">*Filtrate:* add HCl until the liquid is acid, then pass H_2S to saturation, filter and wash quickly, keeping the filter covered with a glass plate; rinse the precipitate into a test-tube, pour upon it some dilute H_2SO_4, boil and filter through a covered filter (note 3):—</td></tr>
<tr><td>*Residue:* dissolve by pouring a little boiling HNO_3 upon the filter, add to the solution AmHO in excess, then $H\bar{A}$ in excess and a few drops of K_4FeCy_6: a chocolate - red precipitate shows :—
Presence of Cu.</td><td>*Filtrate:* add much water and pass H_2S for some time (or add much H_2S-water), a yellow precipitate shows :—
Presence of Cd.
Note.—If this precipitate is dark - coloured, CuS is present (see note 3): filter it off, boil it once more with H_2SO_4; filter and test the filtrate by H_2S as above, for Cd.</td></tr>
</table>

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* If traces of Cu have to be tested for, Na_2S should be substituted for Am_2S, since CuS is

Note 1.—A *white* residue does not necessarily show the absence of Hg.

Note 2.—If the amount of the residue is too small to be removed, the portion of the paper containing it may be cut up and mixed with Na_2CO_3. For the precautions requisite in performing this test see (147).

Note 3.—Unless this precipitate is filtered in a covered funnel, washed quickly, and examined at once, CuS is liable to be oxidised and to pass into the filtrate as $CuSO_4$; it then discolours the CdS precipitate, or may be entirely overlooked if present in small quantity. It is well to pour a few drops of H_2S-water into the funnel with each fresh addition of washing water.

ARSENIC GROUPS.

been well washed with hot water until the last few drops of the
a very slight milkiness on addition of $AgNO_3$, is removed from
filtered :—

GROUP II. B.—ARSENIC GROUP.

The filtrate may contain As_2S_3, Sb_2S_3, SnS_2, SnS, [Au_2S_3, PtS_2]. Add to it HCl until it is acid, and pass H_2S unless the liquid smells strongly; the above sulphides if present will be reprecipitated; a mere milkiness due to precipitation of S may be disregarded.

The precipitate is filtered off and examined as directed in 435 *b* (see note 4, below):—

435 *b.* Remove the precipitate from the filter into a porcelain dish, using as little water as possible; add a little strong HCl and heat, occasionally dropping in a crystal of $KClO_3$, until the precipitate dissolves entirely or leaves only a slight residue of sulphur. Heat this solution just short of boiling (note 5) until it ceases to smell of Cl (note 6), then cool it and pour it, through a filter if necessary, into a small flask (181) containing pieces of pure Zn: if the gas does not come off readily, add a little strong HCl, and cause the gas after passing through some $Pb\overline{A}_2$ solution (see caution 181) to bubble through some $AgNO_3$ solution contained in a broad test-tube. As soon as the evolution of gas slackens, drop in more Zn, unless some remains undissolved in the flask, and as soon as the gas ceases to come off, some Zn still remaining undissolved, remove the test-tube containing the $AgNO_3$ solution; if any black precipitate has formed in this solution it must be examined as below; if the liquid has remained clear and colourless it may be rejected, since As and Sb are absent.

<table>
<tr>
<td rowspan="2">*Residue* in the flask may consist of Sn, (Pt,Au), and excess of Zn; remove any deposit of Sn by shaking the pieces of Zn well with the liquid and quickly pouring off into a dish, decant the liquid and wash the Sn by decantation in the dish: boil the residue with a little strong HCl in a test-tube, then quickly dilute, decant (note 7), and add $HgCl_2$ to the solution; a white precipitate, becoming black with AmHO, shows:—
Presence of Sn (note 8).</td>
<td colspan="2">*To the liquid in the test-tube* add several drops of $AgNO_3$ solution, filter:—</td>
</tr>
<tr>
<td>*Precipitate:* wash well with boiling water until HCl gives no milkiness with the last few drops of the washing-water, then pour upon the filter a little boiling dilute $H_2\overline{T}$ solution, boil it and pour it again through the filter; repeat this several times, then add to the solution HCl and pass H_2S; an orange-red precipitate shows:—
Presence of Sb.
Note.—This precipitate is often only seen on settling from the yellow liquid.</td>
<td>*Filtrate:* dilute several drops of AmHO largely in a test-tube and add this liquid drop by drop to the filtrate: a light-yellow precipitate, which settles in flocks on shaking the liquid, shows:—
Presence of As (note 8).</td>
</tr>
</table>

somewhat soluble in Am_2S; but $Sn''S$ and Au_2S_3 are much more readily soluble in Am_2S.

Note 4.—The detection of As, Sb, Sn by the method given in (435 *b*) is very trustworthy and delicate (208); it is excelled in these respects by the method in (210). For beginners, however, the process (209) is recommended by its greater simplicity.

Note 5.—The solution must not be heated to boiling, else $SbCl_5$ or $SnCl_4$ might be volatilised if present.

Note 6.—In this solution As and Sn will be present as stann*icum* and arsen*icum*, and in this state are not well suited for detection: the following modification makes their detection much more rapid and certain, especially when they are present only in small quantity. After warming until the liquid ceases to smell of Cl, add H_2SO_3 drop by drop, until the liquid after being warmed for a short time smells of SO_2; then heat it short of boiling until the smell of SO_2 is removed, and pour, through a filter if necessary, into the H-apparatus. Great care must be taken to remove the SO_2 entirely, else Sb and As may remain as sulphides in the H-apparatus.

Note 7.—A residue here will contain Au or Pt if they are present: if they are to be tested for refer to (473, 474).

Note 8.—The precipitation by H_2S in the General Table will often show in what condition As or Sn were present (427): further distinctive tests may be tried on the original solution by (187–190) and (201–203).

436. TABLE III.A.—IRON GROUP.

The precipitate produced by boiling, after addition of AmCl and of AmHO in excess, may contain Al_2Ho_6, Cr_2Ho_6, Fe_2Ho_6: the colour of the precipitate will usually show whether it contains any quantity of Fe_2Ho_6 (reddish-brown), or Cr_2Ho_6 (pale green), since Al_2Ho_6 is colourless.

Dissolve the precipitate by pouring upon the filter *a little* boiling dilute HCl, add to the solution pure KHO or NaHO until the precipitate remains after stirring, then add KHO or NaHO in considerable excess, boil for some time and filter:—

<table>
<tr>
<td rowspan="2">1. <i>Filtrate</i> may contain Al_2Ho_6 dissolved in excess of NaHO; if it is green, Cr_2Ho_6 is also in solution and must be precipitated by further boiling in a porcelain dish and then removed by filtration.
Add strong HCl gradually to the liquid drop by drop: a colourless gelatinous precipitate will form, should Al be present. If acid is added in excess this precipitate dissolves, but reappears on adding AmHO in excess. The appearance of this precipitate shows:—
<i>Presence of Al.</i>
<i>Note.</i>—Sodium and potassium hydrates are very liable to contain Al_2Ho_6: the analyst must ascertain that the Al does not come from this source.</td>
<td colspan="2">2. <i>Precipitate</i> may contain Cr_2Ho_6 and Fe_2Ho_6.
Dry upon the filter, carefully avoiding charring the paper; when sufficiently dried the substance will have shrunk considerably into small, hard, dark-coloured pieces; detach these from the filter and fuse them with a little solid Na_2CO_3 and KNO_3 upon a piece of platinum foil, either supported upon a pipeclay triangle or held in the Bunsen flame by crucible tongs. Continue the fusion by heating the under surface of the foil in the blowpipe flame as long as any frothing occurs, then allow to cool; a yellow mass indicates the presence of Cr (note 1). Boil the platinum foil in a small porcelain dish with water until the substance is entirely dissolved or only a dark-brown powder (Fe_2O_3) remains undissolved; take out the foil, allow the powder to settle, and decant the liquid through a filter:—</td>
</tr>
<tr>
<td>3. <i>Filtrate</i> will be yellow if Cr is present (note 1 <i>a</i>).
Add $H\bar{A}$ gradually until it is in excess, which is shown by the colour becoming reddish, boil for several minutes, then add $Pb\bar{A}_2$ solution; a yellow precipitate shows:—
<i>Presence of Cr</i> (note 2).</td>
<td>4. <i>Residue</i> in porcelain dish; dissolve by pouring in a little HCl and boiling, add some water, then a few drops of KCyS solution; a blood-red coloration shows:—
<i>Presence of Fe</i> (note 3).</td>
</tr>
</table>

Note 1.—If Mn was present in the original substance, it is often precipitated more or less completely with the Fe, and will be detected by imparting to the fused mass a bluish-green colour; this should be noted, as the Mn may possibly be entirely precipitated in this group, and will then not be detected in the next group.

Note 1 *a.*—The green colour, due to the presence of Mn, conceals the pale yellow colour due to Cr; but on dissolving the cool mass in water and boiling with $H\bar{A}$, the manganese coloration is destroyed; and, on filtering from any dark-coloured precipitate, the yellow colour due to Cr becomes evident.

Note 2.—Cr may have been present as a chromic salt, Cr replacing H in an acid; or as a chromate, Cr being united with O to form an acid-radicle. Ascertain in which state it was present by boiling some of the original substance with Na_2CO_3 solution and filtering; a yellow filtrate, which on being acidified with $H\bar{A}$ gives a yellow precipitate with $Pb\bar{A}_2$, shows that Cr was *present as a chromate;* Cr, previously combined with an acid as a chromic salt, remains undissolved as green Cr_2Ho_6, and does not colour the filtrate: it is found by 2 in the above Table.

Note 3.—Fe is always detected here in the ferric condition, since, even if originally present as ferrosum, it will have been oxidised by boiling with HNO_3 as directed in the General Table. To ascertain in which condition the Fe was present, boil some of the original substance with HCl, and divide the HCl solution, after filtering, if necessary, into two parts: to one portion add $K_6Fe_2Cy_{12}$, a dark-blue precipitate shows *presence of Fe″* (*Ferrosum*): to another portion add KCyS, a blood-red colour shows *presence of Fe‴* (*Ferricum*).

437. TABLE III.B.—ZINC GROUP.

The precipitate produced by Am_2S in the solution or in the filtrate from Group III.A, may contain ZnS, MnS, NiS, CoS. The colour of the precipitate will usually show whether it contains any quantity of NiS or CoS which are black, or consists only of MnS (pink) or ZnS (white).

The presence of Ni, indicated by the filtrate after boiling with AmHO in the General Table being blue in colour, and also by a dark-brown filtrate being obtained after boiling with Am_2S, will usually have been proved to a certainty by the examination in the borax-bead of the black NiS, precipitated by boiling the dark-brown filtrate from Am_2S (431); if Ni has been thus found its further detection in the Am_2S precipitate, as directed below, will be unnecessary.

Remove the precipitate from the filter with cold dilute HC allow the liquid to stand, occasionally stirring it well. The pre case the milky liquid may be at once examined by 2 (below), or by 1 (below), the filtrate being examined by 2 :—

1. *Residue* (black) may contain NiS, CoS.
Examination by the borax bead.
Fuse a small quantity in a clear borax bead in the extreme tip of the outer blowpipe flame, note its colour; then fuse the bead for some time in the inner blowpipe flame, and again observe its colour. Any one of the following observations may be made :—

Blue bead in both flames shows :— *Presence of Co.* Co need not be further tested for, but if any doubt exists as to the presence of Ni the rest of the residue must be examined as below.	*Brown or yellow bead,* when cold after fusing it in the outer flame, which becomes *grey or opaque* in the inner flame, shows *Presence of Ni, Absence of Co.* Unless mere traces of Co have to be tested for, no further examination need be made.	*A bead which is neither brown nor blue,* but of some intermediate hue on cooling from the outer flame, indicates the probable presence of both Co and Ni. Examine the remainder of the black residue as directed below for Ni and Co, or for Co alone if Ni has been already detected by (431).

Further examination of the residue.—Rinse the residue from the filter into a porcelain dish, using as little water as possible: pour in a little strong HCl and boil for some time, adding occasionally a small crystal of $KClO_3$, until the black residue is entirely dissolved or only a small quantity of dark sulphur remains; then boil down nearly to dryness, a blue liquid shows *presence of Co*; dilute with a little water, filter if necessary into a boiling-tube and pour in KCy solution slowly until the precipitate formed at first is just redissolved, boil briskly for several minutes, and add much strong Na_2Cl_2O, or make decidedly alkaline with NaHO and add much Br-water; heat nearly to boiling, and allow the liquid to stand at least ten minutes, filter (note 1, page 237) :—

3. *Precipitate* (*black*): wash, and confirm the presence of Ni by fusing some of the precipitate, or the paper stained with the precipitate, in a borax bead in the outer and inner blowpipe flames: a bead *yellow* when cold, becoming *black* in the inner flame shows :— *Presence of Ni.*	4. *Filtrate* must be warmed with more Na_2Cl_2O or Br-water, and filtered from any additional precipitate which may form, then evaporated to dryness and strong HNO_3 poured upon the residue as long as any frothing is caused; this is then evaporated just to dryness, the residue dissolved in water, excess of KHO added to it, and any precipitate filtered off and fused into a borax bead, taking the paper stained by the precipitate if the precipitate is not easily removed: a blue bead shows :— *Presence of Co.*

438. TABLE IV.—BARIUM GROUP.

The precipitate produced by Am_2CO_3 may contain $BaCO_3$, $SrCO_3$, $CaCO_3$: it is to be well washed with boiling water. Before dissolving the precipitate off the filter much may usually be learnt by an examination of the flame coloration yielded by the HCl solution (see 1 below). The results thus obtained are afterwards confirmed in the wet way (see 2 below).

in the way described in par. 26 *c*; take out the paper and
cipitate will either dissolve, leaving only white sulphur, in which
black residue will be left which must be filtered off and examined

2. *Solution or filtrate* may contain Zn, Mn.
Boil in a porcelain dish until it ceases to smell of H_2S, then drop in a small crystal of $KClO_3$, and boil for several minutes. After cooling the liquid add pure NaHO until after stirring the liquid it turns red litmus-paper blue, then add more NaHO, stir well and filter:

5. *Precipitate is white at first, but rapidly darkens* in the air. Fuse a portion of it with solid Na_2CO_3 and KNO_3 on platinum-foil; a bluish-green mass is obtained on cooling, showing: *Presence of Mn.* *Note.*—Mn may have been originally present either replacing H in an acid, or combined with O as an acid-radicle. Its presence in an acid-radicle is indicated by the colour of the original solution, since in the form of manganate it is green, and as permanganate it is purple; these colours disappear on boiling the HCl solution for some time, or on passing H_2S.	6. *Filtrate;* pass H_2S, a white precipitate forms, often somewhat discoloured, showing: *Presence of Zn.* *Note.*—From a dilute solution of $ZnHo_2$ in NaHO the $ZnHo_2$ is precipitated on boiling; hence after adding NaHO, as directed above, the liquid must not be boiled, else Zn might be precipitated with the $MnHo_2$ and escape detection. But by diluting and boiling the above filtrate it is usually possible to cause any $ZnHo_2$ it holds in solution to precipitate, especially if the alkalinity of the liquid be somewhat reduced by cautious addition of HCl in quantity insufficient to make the liquid acid. If this precipitate be separated by pouring the liquid through a double filter and then be dissolved off the filter and the sides of the boiling tube by a little boiling $H\bar{A}$, ZnS may be precipitated *perfectly white* from this solution by H_2S, showing: *Presence of Zn.*

Note 1.—If this precipitate adheres to the sides of the boiling tube, it is dissolved, after the liquid has been emptied out, by pouring in a little boiling HCl; from this solution the Ni is precipitated by addition of KHO, and the precipitate filtered off and tested by the borax bead for Ni.

1. *Examination by flame coloration.*—Take a small quantity of the precipitate off the filter upon the end of a glass rod, and dissolve it by moving the end of the rod about in several drops of HCl on a watch glass; dip into this solution a loop of platinum wire which imparts no colour to the flame, then hold the loop for some time in the Bunsen flame, repeating the process if the coloration is not satisfactorily observed at first. Ca will impart to the flame

a *yellowish-red* colour, Sr a *crimson-red*, Ba a *yellowish-green*. The
ing the flame through the indigo-prism; the Ca coloration then
Ba is usually found by its coloration remaining after the others
their colorations are apt to interfere with one another; but if at
tainly present; if a green coloration is visible without using the

2. *Examination in the wet way.*—The rest of the precipitate is
again boiling the liquid, and pouring it once more through the
the solution is acid, and if it is not, add $H\bar{A}$ whilst stirring until
solution into a test-tube, and put by the larger portion (*B*). To
part of the tube in cold water or holding it in a stream from the
cipitate forms refer to Column I. (below); if no immediate precipi-
forms refer to Column II., if no precipitate forms even after

<table>
<tr><td colspan="3">I. <i>An immediate precipitate is produced by $CaSO_4$ in the cold, showing Presence of Ba.</i>
Portion (<i>B</i>) must then be tested for Sr and Ca which may also be present: examine it as directed below :—
Add K_2CrO_4 solution until the colour of the liquid is reddish-yellow; heat, and filter through a double filter-paper, pouring the filtrate through the same filter repeatedly if necessary until the liquid is quite clear; then add to the liquid, which must be orange-red in colour, AmHO until the colour changes to pale yellow, then add Am_2CO_3 solution :—</td></tr>
<tr><td rowspan="2"><i>No precipitate forms,</i> showing :—
<i>Absence of Sr and Ca.</i></td><td colspan="2"><i>A precipitate forms,</i> showing Sr, Ca, or both of them, to be present. Add to the liquid Am_2CO_3 in excess, filter, reject the filtrate and dissolve the precipitate off the filter in as little boiling $H\bar{A}$ as possible; pour off a small part (<i>A'</i>) of this solution, reserving the larger portion (<i>B'</i>). To (<i>A'</i>) add $CaSO_4$ solution and boil :—</td></tr>
<tr><td><i>No precipitate forms :—</i>
<i>Absence of Sr.</i>
Examine portion (<i>B'</i>) for Ca as directed at (<i>C</i>) in Column III.</td><td><i>A precipitate forms :—</i>
<i>Presence of Sr.</i>
Examine portion (<i>B'</i>) for Ca as directed at (<i>C</i>), Column II.</td></tr>
</table>

439. TABLE V.—

The filtrate, after addition to the original solution of all the group-
Since, however, NH_4-salts have been added as group-reagents, it is
boiling a portion of the original substance with KHO solution, and
cognised by its smell or its action on moistened red litmus-paper.
it will have been already tested for in the preliminary examina-

red colorations yielded by Ca and Sr are distinguished by view- appears *dingy-green,* whilst the Sr coloration still appears *crimson ;* have disappeared. When all three metals are present together any time a red colour is visible through the indigo-prism, Sr is cer- prism, the presence of Ba is proved.

dissolved off the filter by pouring upon it a little boiling $H\bar{A}$, filter if the precipitate is not entirely dissolved. Test whether the liquid is acid, then pour off a small portion (*A*) of the acid the portion (*A*), after it has been cooled by immersing the lower tap, add several drops of $CaSO_4$ solution; if an immediate pre- tate forms heat the liquid to boiling, and if a precipitate then several minutes refer to Column III.

II. *A precipitate is not formed at once on the addition of $CaSO_4$, but appears on boiling the liquid, showing :—* *Absence of Ba and Presence of Sr.* *C.* Portion (*B*) is then examined for Ca as directed below :— Add dilute H_2SO_4 in excess, boil and filter, reject the precipitate ; add to the filtrate several drops more H_2SO_4 and boil ; if this causes any precipitate boil and filter, and again test the filtrate by addition of H_2SO_4 and boiling : repeat this process if necessary. To the clear filtrate, which gives no further precipitate on addition of H_2SO_4 and boiling, add gradually AmHO until after mixing the liquid by thorough stirring or shaking, it turns red litmus-paper blue, then add $Am_2C_2O_4$ solution and warm gently ; a white precipitate, often appearing only after a time, shows :— *Presence of Ca.* (See Note, Column III.)	III. *No precipitate is produced by $CaSO_4$, even on boiling, showing :—* *Absence of Ba and Sr, and Presence of Ca.* *C.* Confirm the presence of Ca by making portion (*B*) alkaline with AmHO (35 *a*), then add $Am_2C_2O_4$ solution ; a white precipitate forms, showing :— *Presence of Ca.* (See note below.) *Note.*—If mere traces of Ca have to be tested for, the filter-paper used in the analysis must first be freed from any traces of Ca it may contain by wetting it with dilute HCl, and then thoroughly washing it with distilled water, as described in the note to par. 23*a*(p. 45).

POTASSIUM GROUP.

reagents in succession, may still contain Mg, K, Na, and NH_4. useless to test for NH_4 in this filtrate : NH_4 must be detected by ascertaining whether any NH_3 gas is evolved, which would be re- It is, however, usually unnecessary to test here for NH_4, since tion (390). Proceed to examine for Group V. as directed below.

Evaporate the filtrate from Group IV. to dryness in a porcelain dish, scrape out the solid residue and heat it to redness upon a piece of platinum foil in the Bunsen flame as long as any white fumes are seen to be given off on removing the foil for an instant from the flame (note 1). All NH_4-compounds are thus entirely removed. If any residue remains (note 2) it is to be dissolved by boiling the foil in a test-tube with a small quantity of water to which several drops of dilute HCl have been added. Divide this solution into two equal parts :—

<table>
<tr>
<td rowspan="3">EXAMINATION FOR Mg.
Before testing for Mg in one portion of this solution it is necessary to remove from it any traces of Ba, Sr, or Ca which it may possibly contain*, and which might else be mistaken for Mg.
Add, therefore, several drops of H_2SO_4 to the liquid, boil for a short time and let stand; then, whether a precipitate has formed or not, add a little AmCl, then AmHO in excess, then several drops of $Am_2C_2O_4$, and warm gently; if any precipitate has formed proceed to filter at once as quickly as possible. To the liquid in which the addition of H_2SO_4, AmCl, AmHO in excess, and $Am_2C_2O_4$ causes no further precipitate add Na_2HPO_4, and if no precipitate forms at once warm gently, shake or stir the liquid violently and let it stand for some time: a white crystalline precipitate shows :—
Presence of Mg.</td>
<td colspan="2">EXAMINATION FOR K AND Na.
In the other portion proceed to test for K and Na as directed below. The presence of Mg in no way interferes with the detection of K and Na, and hence if it has been detected its removal is unnecessary.
Flame coloration.—Dip into the solution a loop of platinum wire which has been proved when held in a Bunsen flame not to impart any colour to it. Hold the loop in the Bunsen flame; one of the following results will be observed :—</td>
</tr>
<tr>
<td>*A bright-yellow flame coloration*, indicating the *presence of Na* (note 3).
Examine this coloration through the indigo-prism; it appears red, indicating *presence of K:* if no red colour is visible K is probably absent or present only in very minute quantity.</td>
<td>*A pale violet coloration*, appearing crimson red through the indigo-prism, shows :—
Presence of K, absence of Na.</td>
</tr>
<tr>
<td colspan="2">It is usual to confirm the results of the examination of the flame coloration by pouring the remainder of the solution upon a watch glass, adding to it several drops of $PtCl_4$ (note 4) and stirring well for some time; the formation of a yellow precipitate shows :—
Presence of K.
See "Remarks (47)," and Note 5, page 241.</td>
</tr>
</table>

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* Traces of Ba, Sr, Ca may remain unprecipitated by Am_2CO_3 in Group IV.

Note 1.—If the residue is small in amount it may be ignited in the dish; but this is not to be generally recommended, as the porcelain dish is liable to be cracked by the heat, and it is also difficult to get entirely rid of the NH_4-salts by heating only in porcelain.

Note 2.—It is not safe to place much reliance upon an examination of the foil for the detection of a small quantity of residue, but it may usually be detected by its producing a crackling noise whilst the foil is cooling immediately after its removal from the flame. Should there be any doubt, the foil must be boiled with water and a drop of HCl, and the solution examined for Mg, K, and Na, as directed above.

Note 3.—A yellow coloration, more or less intense, will almost always be obtained here, since nearly all substances and reagents contain small quantities of Na; hence the student must note the intensity of the coloration, and judge from it whether the quantity of Na is small or large; he must then enter accordingly, either "*Presence of Na,*" or "*Presence of trace of Na.*"

Note 4.—If iodine is present, $PtCl_4$ will produce an intense red coloration; hence if iodine is suspected to be present, before adding $PtCl_4$ the above solution should be evaporated to dryness with a little strong HNO_3, the residue dissolved in a few drops of dilute HCl and tested with $PtCl_4$ for K. This is of course unnecessary if, before precipitating Group III. in the General Table, the solution has been evaporated to dryness with HNO_3. In case of uncertainty, add $PtCl_4$ to a drop only of the above solution; and if iodine is found to be present proceed as directed above.

Note 5.—Mere traces of K and Na may be detected by adding $PtCl_4$, and evaporating the liquid to dryness in a porcelain dish upon a water-bath (504); then pouring absolute alcohol into the dish and stirring; any yellow residue shows *presence of K*, the solution giving the pure Na coloration if Na is present. By filtering off the yellow residue and washing it with absolute alcohol, it will yield the pure K flame coloration.

EXAMINATION FOR ACID-RADICLES.

440. Several acid-radicles will probably have been already detected for certain, if present, by the preliminary examinations. Such are CO_3, SO_3, S, NO_2, ClO, NO_3, ClO_3, $\bar{A}$: their reactions are so characteristic, that unless interfered with by the presence of other substances, their presence or absence will have been proved to a certainty: in case any uncertainty is felt, the analyst should refer to the reactions given in Section IV. for the particular acid-radicle in question and select a reaction which will be decisive.

The examination for metals also frequently yields proof of the presence of certain acid-radicles. Thus on passing H_2S into the HCl solution:—

A reddish-yellow solution, becoming green and depositing white sulphur, shows	*Presence of CrO_4*
A green solution, becoming colourless ,,	*Presence of $(MnO_4)''$*
A purple solution ,, ,, ,,	*Presence of $(MnO_4)'$*
A yellow precipitate of As_2S_3, appearing only when the liquid is boiled, shows	*Presence of AsO_4*
An insoluble residue of SiO_2, after evaporating to dryness the filtrate from the H_2S-group, shows:	*Presence of SiO_3*
And a yellow precipitate obtained with $AmHMoO_4$ before precipitating Group III., shows:—	*Presence of PO_4*

The presence of any of these acid-radicles thus detected will require no further confirmation.

441. Of the acid-radicles which remain to be tested for, some can be detected by the plan drawn out below (442–445); others are best detected by special tests made on the original substance (446–454).

Much time and trouble may usually be saved by considering what acid-radicles can possibly be present. A careful preliminary examination will usually have limited this

443.	1. *Portion acidified with HCl.*	*Present.*
	On addition of $BaCl_2$ solution a white precipitate insoluble on boiling (444, 1) . . , . .	$(SO_4)''$
	On addition of $BaCl_2$ solution, a semi-transparent precipitate insoluble on boiling (444, 2) . .	$(SiF_6)''$
	On addition of AmCl and Am_2CO_3, a semi-transparent precipitate	$(SiO_3)''$
	On addition of $FeSO_4$ solution, a dark-blue precipitate	$(Fe_2Cy_{12})^{vi}$
	On addition of Fe_2Cl_6 solution, a dark-blue precipitate: $FeSO_4$ yielding a light-blue precipitate .	$(FeCy_6)^{iv}$
	On addition of Fe_2Cl_6 solution, a red coloration, destroyed by pouring into $HgCl_2$ solution . .	$(CyS)'$

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* F will be readily detected in this precipitate, or bett

number considerably, and it may be further reduced by the knowledge already obtained as to the solubility of the substance under analysis, and the metals it contains. A reference to the Table of Solubilities (455), and the explanatory paragraphs 456, 457, and 458 will show how this knowledge is applied.

General Examination for certain Acid-radicles.

442. Before employing the liquid tests it is advisable to separate from the substance any metals other than K, Na, and NH_4 which it may contain, since some of these are liable to be precipitated by the reagents added for the detection of the acid-radicles. If alkali-metals alone are present this separation is unnecessary, since they are not precipitated by any of the reagents.

This separation of the metals may usually be effected by boiling a portion of the substance with Na_2CO_3 solution, which must be added as long as it causes any precipitate. Filter from the precipitate, and divide the clear filtrate into five equal portions. Reserve one of these portions in case of accident and a second for the tests for organic acid-radicles, and acidify the others whilst they are hot by addition of HCl, HNO_3, and $H\bar{A}$ respectively: examine these portions as directed below, using a separate part for each test:—

II. *Portion acidified with* HNO_3 Refer to (444, 3).	*Present.*	III. *Portion acidified with* $H\bar{A}$	*Present.*
On addition of $AgNO_3$, a *pure white* precipitate, easily soluble in AmHO (444, 1) . .	Cl'	On addition of $Pb\bar{A}_2$ solution a yellow precipitate . .	$(CrO_4)''$
On addition of $AgNO_3$, a *light-yellow* precipitate, with difficulty soluble in AmHO .	Br'	On addition of $CaCl_2$ solution a white gelatinous precipitate	F', probably.
On addition of $AgNO_3$, a *yellow* precipitate, almost insoluble in AmHO . . .	I'	On addition of $CaCl_2$ a white pulverulent precipitate .	$(C_2O_4)''$*, probably.
Refer to (445).		On addition of Fe_2Cl_6 a yellowish-white precipitate (449, 450).	$(PO_4)'''$, or $(AsO_4)'''$.

n the original substance by (452), $(C_2O_4)''$ by (453).

NOTES ON THE PRECEDING TABLE.

444. 1. Unless the Na_2CO_3 solution used in preparing the solution for these tests was pure, $(SO_4)''$ and $(Cl)'$, if detected, may have been present only as impurities in the Na_2CO_3: portions of the original substance should then be tested by (446) and (447).

2. The presence of $(SiF_6)''$ should be confirmed by other tests, such as adding KCl (300), or evolving HF by strong H_2SO_4 (301); the HF is most readily evolved from the $BaSiF_6$ precipitate (301 a).

3. Should $AgNO_3$ yield a black precipitate, this proves the presence of a sulphide, or possibly a theiosulphate; add HNO_3, and boil; the black Ag_2S will thus be decomposed, leaving a milky liquid, in which any other precipitate is readily seen after being coagulated by heating or shaking.

445. It must be remembered that $(Cy)'$, $(FeCy_6)^{iv}$, $(Fe_2Cy_{12})^{vi}$, and (CyS) are also precipitated by $AgNO_3$, and therefore if these acid-radicles have been already found, a precipitate produced by $AgNO_3$ does not prove the presence of $(Cl)'$, $(Br)'$, or $(I)'$, which acid-radicles must be specially examined for, as directed below.

If chloride, bromide, and iodide have all to be tested for, a portion of the Na_2CO_3 solution must be examined by (273); or the precipitate obtained by $AgNO_3$ (see 443, II.) may be tested by (273 *a*) for Cl, Br, I.

If only bromide and iodide have to be tested for, use par. 274.

SPECIAL TESTS FOR ACID-RADICLES TO BE MADE ON THE ORIGINAL SUBSTANCE.

446. *Sulphate.*—A portion of the original substance is boiled with HCl, the liquid decanted or filtered if not clear, and $BaCl_2$ added to it: a white precipitate shows *presence of SO_4.*

447. *Chloride.*—A portion of the original substance is warmed with HNO_3, the solution decanted or filtered if necessary, and $AgNO_3$ solution added to it, a *perfectly white* precipitate, easily dissolved by warm AmHO, shows *presence of chloride.*

448. *Cyanide* if present will have been detected by its special test and by the smell of bitter almonds, which is given off by the substance, after adding H_2SO_4 (411). Add to some of the substance Am_2S in excess and boil; filter from any dark-coloured precipitate, boil the filtrate for a short time, acidify with HCl and add Fe_2Cl_6; a red coloration, not destroyed by heat but removed by addition to $HgCl_2$, shows *presence of Cy*, possibly as $(FeCy_6)^{iv}$, $(Fe_2Cy_{12})^{vi}$, or (CyS), if these acid-radicles have been found (443, 1).

449. *Arsenate.*—This acid-radicle cannot be present unless As was detected during the examination for metals; the presence of $(AsO_4)'''$ is there rendered probable by the precipitation of yellow As_2S_3 occurring only when the liquid saturated with H_2S is boiled. If As has been found amongst the metals, proceed to test for $(AsO_4)'''$ by adding to a portion of the Na_2CO_3 solution (442) HCl until it is acid, then AmCl, AmHO in excess, and $MgSO_4$; filter off any precipitate which forms on warming and shaking the liquid, and pour a few drops of $AgNO_3$ solution upon the white precipitate on the filter, a change of colour to brown shows *presence of* $(AsO_4)'''$.

450. *Phosphate.*—Boil some of the original substance with dilute HNO_3, add a little of this clear solution, filtered if necessary, to some $AmHMoO_4$ solution, shake and stir the liquid well, and if no precipitate forms warm *very gently*, a yellow precipitate shows *presence of* $(PO_4)'''$.

If $(AsO_4)'''$ has been detected (449) this test for $(PO_4)'''$ is only trustworthy when the yellow precipitate has been obtained either in the cold or by employing a *very gentle* heat. If any doubt is felt concerning the presence of $(PO_4)'''$, boil some of the substance with strong HCl, and examine for $(PO_4)'''$ after having entirely separated the $(AsO_4)'''$ by passing H_2S into the boiling HCl solution. A doubtful precipitate with $AmHMoO_4$ may be proved to contain phosphate by dissolving it in AmHO, adding AmCl and $MgSO_4$, filtering and dropping $AgNO_3$ upon the precipitate, if phosphate is present it turns *yellow*, if arsenate *brown*.

451. *Borate.*—Warm a portion of the substance with a little dilute HCl, dip into the solution a strip of turmeric-paper and dry it in a steam-oven or at a gentle heat, if the slip is *reddish-brown* and becomes *blue-black* when moistened with AmHO, *the presence of* $(BO_3)'''$ is shown.

452. *Fluoride.*—Pour upon a portion of the powdered substance strong H_2SO_4; and warm the mixture in a leaden or platinum crucible covered with a watch glass, which has been coated with wax or paraffin, and has then had characters traced through the film with the point of a penknife:

the characters are etched upon the glass showing *the presence of F.*

If SiO_2 is known to be present, the test must be made by conducting the gas, evolved on heating the substance with strong H_2SO_4, into dilute AmHO, when a deposit of gelatinous H_4SiO_4 proves the *presence of F.*

453. *Oxalate.*—The precipitate produced by $CaCl_2$ in III. (443), or the original substance if it contains no carbonate, is mixed on a watch glass with MnO_2 free from carbonate, and strong H_2SO_4, and gently warmed: CO_2 is evolved and detected by holding over the bubbles which rise from the mixture a rod moistened with lime water, or by inverting another watch glass containing on its under surface a drop of lime water over the glass containing the mixture.

453 *a. Tartrate.*—The presence of $\overline{T}$ will probably have been indicated in the Preliminary Examination (388 or 412). In order to confirm its presence, any metals of Groups I., II., and III. must be separated by H_2S or Am_2S, and $Ca\overline{T}$ is then precipitated from the liquid, made just alkaline with AmHO and mixed with a little AmCl, by

455. TABLE SHOWING THE SOLUBILITY OF

A blank signifies that the solubility

		K.	*Na.*	*Am.*	*Mg.*	*Ba.*	*Sr.*	*Ca.*	*Fe''.*	*Fe'''.*	*Al.*	*Cr.*	*Zn.*	*Mn.*	*Ni.*
1.	Oxide . .	w.	w.	w.	a.	w.	w.	w-a.	a.	a.	a.i.	a.i.	a.	a.	a.
2.	Sulphide .	w.	w.	w.	w-a.	w.	w.	w-a.	$a^{m.}$	a.	—	—	a.	a.	$a^{mn.}$
3.	Chloride .	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	a.i.	w.	w.	w.
4.	Iodide .	w.	w.	w.	w.	w.	w.	w.	w.	w.	—	—	w.	w.	—
5.	Sulphate .	w.	w.	w.	w.	i.	i.	$w\text{-}a^{m}\text{-}i.$	w.	w.	w.	w.a.	w.	w.	w.
6.	Nitrate .	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.
7.	Phosphate	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.
8.	Carbonate	w.	w.	w.	a.	a.	a.	a.	a.	—	—	—	a.	a.	a.
9.	Borate .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.
10.	Arsenite .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.
11.	Arsenate .	w.	w.	w.	a.	a.	a.	a.	a.	a.	—	—	—	—	a.
12.	Chromate .	w.	w.	w.	w.	a.	a.	a.	—	w.	a.	a.	w.	i.	a.
13.	Fluoride .	w.	w.	w.	a.	a-i.	i.	a-i.	—	—	—	—	—	—	—
14.	Oxalate .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	w-a.	a.	w-a.	a.

Only the most commonly occurring compounds are contained in this table, the solubility of other substances may be obtained by reference to Storer's "Dictionary of Solubilities."

456. The table is thus arranged: in a horizontal line at the head are adding excess of $CaCl_2$, shaking well and letting stand for some time. A precipitate may consist of $Ca\overline{T}$, $Ca_3(PO_4)_2$, or $Ca\overline{O}$. By shaking this precipitate with cold KHO solution, $Ca\overline{T}$ is dissolved and may be reprecipitated from the solution or filtrate by diluting and boiling for some time: the liquid is decanted and the precipitate is then gently warmed with a little *very dilute* AmHO and a crystal of $AgNO_3$, a metallic mirror shows the *presence of* $\overline{T}$.

454. *Sulphide.*—Very small quantities of S may be detected by boiling the substance with KHO for some time, and adding to the clear solution alkaline $Pb\overline{A}_2$ solution, when a black precipitate or coloration will appear.

The above directions must only be considered as affording examples of how the principle acid-radicles are detected. By reference to the individual reactions in Section IV. and to the directions there given for the detection of similar acid-radicles when occurring together, this method will serve for the detection of all the commonly occurring acid-radicles.

SUBSTANCES IN WATER AND ACIDS.

is unknown or unimportant.

Co.	Hg''.	*Pb.*	*Bi.*	*Cu.*	*Cd.*	*Sb.*	Sn''.	Sn''''.	As'''.	*Ag.*	Hg'.		
a.	a.	a^n.	a.	a.	a.	a^m.	a.	a.i.	w.a^m.	a^n.	a^n.	O.	1.
a^{mn}.	a^{mn}.	a^n.	a^n.	a^n.	a.	a^m.	a^m.	a^m.	a^n.	a^n.	a^n.	S.	2.
w.	w.	w.i.	w.b.	w.	w.	w.b.	w.b.	w.	w.	i.	a.i.	Cl.	3.
—	a.	w.a^m.	—	—	w.	—	w.	w.	—	i.	a^n.	I.	4.
w.	w.b.	a.i.	w.b.	w.	w.	a.	w.	w.b.	—	w.a^n.	wb.a^n.	SO_4.	5.
w.	w.b	w.	w.b.	w.	w.	—	—	w.	—	w.	w.b.	NO_3.	6.
a.	a.	a^n.	—	a.	a.	—	a.	—	—	a^n.	a^n.	PO_4.	7.
a.	a.	a^n.	a.	a.	a.	—	—	—	—	a^n.	a^n.	CO_3.	8.
a.	—	a^n.	a.	a.	w.a.	—	a.	—	—	—	w.	BO_2.	9.
a.	a.	a^n.	a.	a.	—	a.	—	—	—	a^n.	a^n.	AsO_2.	10.
a.	a.	a^n.	—	a.	—	a.	—	—	—	a^n.	a^n.	AsO_4.	11.
—	w-a.	a^n.i.	a.	w.	—	a.	—	—	—	a^n.	a^n.	CrO_4.	12.
—	w.	a.	—	—	—	—	—	—	—	w.	—	F.	13.
a.	a.	a.	a.	a.	a.	a.	a.	w.	—	a.	a.	C_2O_4.	14.

placed the more commonly occurring metals, which yield salts or basic oxides: in the vertical column on the left is a list of that portion of the names of the compounds thus formed, which corresponds to the acid-radicle: the same are also placed in chemical symbols in a vertical column on the right. The solubility of a compound is denoted by letters:—

w.	Signifies soluble in water.
a.	Soluble in acids; the term standing for HCl, HNO_3, and aqua regia.
a^m.	Soluble in muriatic, or hydrochloric acid.
a^n.	Soluble in nitric acid.
am^n.	Soluble in a mixture of muriatic and nitric acids, or aqua regia, but not in either separately.
i.	Insoluble in water and acids.
w.a.	Letters thus placed together with a stop between, signify that in different states the substance shows these different solubilities.
w-a.	Letters connected by a hyphen indicate that the substance is only slightly soluble in the first solvent, and may therefore partially fall under the class denoted by the second letter.
w.b.	Decomposed more or less by much water with formation of a basic salt which is insoluble in water but soluble in acid.

457. To find the solubility of any compound of one of the metals placed at the top of the Table, glance down the vertical column which is headed by this element, the letter indicating the solubility of the compound will be found in a horizontal line with the acid-radicle portion of the name. Thus to find the solubility of zinc sulphate, it is only necessary to glance down the vertical column with Zn at its head: on a horizontal line with "sulphate" (SO_4) stands the letter *w*, showing the zinc sulphate is soluble in water.

458. The way in which this Table is used after ascertaining the solubility of a substance under analysis, and detecting the metals present in it, may be explained by an example. In a substance *which was entirely soluble in water*, the metals found were K, Ba, Ag. We glance down the columns headed by these three metals, and can thus see at once which acid-radicles may be present: all acid-radicles might be present combined with K since all its salts are soluble in water, but the presence of Ba in a substance soluble in water shows that SO_4, PO_4, CO_3, BO_3, AsO_3, AsO_4, CrO_4, and F cannot be present, since these acid-radicles form compounds with Ba which are insoluble in water: Ag excludes in addition S, Cl, Br, and I; hence amongst the more commonly occurring acid-radicles only NO_3 need be tested for.

This example shows how much we may often simplify the examination for acid-radicles, when the metals present in a substance and also its solubility are known, by referring to the above Table.

REMARKS ON THE PRECIPITATION OF GROUP III.

459. In the General Table (419) it is assumed that in the absence of $(PO_4)'''$ the two Sub-groups III.A and III.B can be separated from one another by adding first AmCl and then excess of AmHO to the solution; Group III.A alone being thus precipitated, and Group III.B being afterwards precipitated by adding Am_2S to the filtrate. Now although AmCl entirely prevents the precipitation of Group III.B by AmHO, if the solution is kept covered from the air; still if the members of Group III.A are present at the same time in the solution they are precipitated by AmHO, and the presence of AmCl will not prevent Mn and Zn from being precipitated with them in smaller or larger quantity, Mn particularly showing a tendency to be precipitated with Fe, and Zn with Cr. Hence if either Mn or Zn, especially the former, be present in small quantity only, it may be entirely precipitated in Group III.A: this is not a serious matter in the case of Mn, since it is readily detected in the ordinary examination of the precipitate by Table III.A by yielding a *green* mass on fusion with Na_2CO_3 and KNO_3; but Zn may be entirely passed over, since if precipitated with Cr_2Ho_6 its presence would certainly not be detected in Table III.A. The method of dissolving the precipitate several times in HCl and reprecipitating with AmHO, to some extent meets this difficulty; but it is preferable whenever a precipitate is produced on adding AmCl and AmHO, and traces of Mn and more particularly of Zn have to be tested for, to precipitate Groups III.A and III.B together, by adding in succession AmCl, AmHO, and Am_2S, and boiling. The precipitate is then examined by Table III.C (464, 465) for Groups III.A and III.B, the filtrate being examined, as directed in the General Table, for Groups IV. and V. It must, however, be understood that if no precipitate is produced by AmCl and AmHO, Am_2S may be added, and the precipitate, if any, examined by Table III.B, since in the absence of Group III.A the members of Group III.B. are not precipitated by AmHO in the presence of AmCl.

460. *The presence of* $(PO_4)'''$ *in the HCl solution* which is to be examined for Groups III., IV., and V. involves no special procedure if AmHO added after AmCl produces no precipitate, since the phosphates of Groups III. and IV. and of Mg must be absent. If, however, on adding AmCl and AmHO a precipitate is formed, the directions given at the head of Table III.D (466) must be followed, and the precipitate must be examined by that Table. The reason for this departure from the ordinary course of analysis is that, whereas Al, Cr, Ba, Sr, Ca and Mg, if present as phosphates, are completely precipitated by AmHO, the phosphates of Ni, Co, Mn, Zn and Fe, are only partially precipitated by AmHO; their metals are however entirely precipitated by Am_2S. The filtrate from Am_2S is then examined as directed in the General Table for Groups IV and V.

The principles on which the method drawn out in Table III.D. is founded are:—

1. The insolubility of the phosphates of Al, Fe and Cr in $H\bar{A}$ in the presence of an alkaline acetate, the other portions of the precipitate being soluble.

2. The separation of all the $(PO_4)'''$ which is in the $H\bar{A}$ solution combined with Ba, Sr, Ca, or Mg, by Fe_2Cl_6 in an $H\bar{A}$ solution.

The further separation of Al, Fe and Cr phosphates is somewhat complicated by the fact that $AlPO_4$ is only decomposed by fusion mixture when SiO_2 is also present, and this SiO_2 has to be removed after the fusion by methods which will be intelligible on reference to (290, 291).

461. The reason why, in obtaining the precipitate for Table III.D, the precipitates produced by AmHO and Am_2S are filtered and washed separately, is that phosphates of Fe, Zn, Mn, Ni, and Co are converted by Am_2S into sulphides, forming at the same time ammonium phosphate which remains in solution, and this would precipitate Ba, Sr, Ca, Mg as phosphates from the solution even if they were not originally present in that condition. Now this besides complicating the process of analysis, would also render it impossible to state whether Ba, Sr, Ca and Mg were originally present as phosphates or not. Hence the phosphates of Groups III. and IV. and of Mg are first precipitated by AmCl and AmHO, then any members of Group III.B and any remaining traces of phosphates of Group III. are precipitated from the filtrate by Am_2S. The two precipitates are mixed and treated with Am_2S, which will dissolve away the $(PO_4)'''$ from Fe, Zn, Mn, Ni, Co phosphates if present, leaving the other phosphates undecomposed; hence, if on filtering and testing the filtrate with AmCl, AmHO and $MgSO_4$ a white crystalline precipitate is obtained, this indicates the presence of $(PO_4)'''$ and indirectly establishes the presence in the original precipitate of phosphate of some one or more of the metals Fe, Zn, Mn, Ni, Co.

462. Oxalates, borates, fluorides, and silicates of Ba, Sr, Ca, Mg would likewise be precipitated by AmHO in Group III.A; but by the evaporation of the HCl solution after passing H_2S, H_3BO_3 and HF are usually volatilised, and H_2SiO_3 becomes insoluble; oxalates are also decomposed by a gentle ignition after evaporation.

⁂ The best course to be pursued in precipitating and examining Group III. will be found in (463).

463. RULES FOR THE PRECIPITATION AND EXAMINATION OF GROUPS III.A AND III.B.

Refer to para. 459–462 for the explanation of these Rules.

The following rules may be laid down for precipitating and detecting members of Groups III.A and III.B in the HCl solution, a small portion of which has been tested for $(PO_4)'''$ by $AmHMoO_4$:—

I. *If the addition of AmHO after AmCl causes no precipitate*, shewing the absence of Al, Fe, Cr and of their phosphates, and of phosphates of Ba, Sr, Ca and Mg, Am_2S is at once added, the liquid boiled, and the precipitate, if any, examined for members of Group III.B by Table III.B. (437): this course is pursued whether $(PO_4)'''$ is present or absent, since even if phosphate is present, metals of Groups III. and IV. and Mg are not present as phosphates.

II. *If the addition of AmHO after AmCl causes a precipitate, shewing the presence of Al, Fe, Cr, and $(PO_4)'''$ is not present*, the plan of precipitation depends upon the object of the analysis.

If small quantities of Zn need not be tested for, the method given in the General Table (419) may be followed.

If traces of Zn have to be tested for in the solution, add AmCl, then AmHO and Am_2S in excess, boil, filter, and examine the precipitate by Table III.c_1 (464) or III.c_2 (465). The method described in Table III.c_1 gives trustworthy results, and is to be used for very careful analysis; the method in Table III.c_2 is however much more simple, and is sufficiently accurate for general use.

III. *If the addition of AmHO after AmCl causes a precipitate, and $(PO_4)'''$ is present*, the method of precipitation to be adopted will be found in par. 466, together with the Table for the examination of the Group precipitate.

464. TABLE III.c_1.—THE SEPARATION OF GROUPS III.A AND III.B BY $BaCO_3$.

The precipitate may contain Fe, Al, Cr, Zn, Mn, Ni, Co. Rinse it off the filter into a porcelain dish, using as little water as possible, add some strong HCl and boil, adding at intervals a small crystal of $KClO_3$ until all is dissolved but a small quantity of yellow sulphur. Evaporate very nearly to dryness, dilute with a little water, and pour, through a filter if necessary, into a small flask. Cool, pour in a small quantity of $BaCO_3$ suspended in water, cork the flask tightly and shake well; repeat the addition of $BaCO_3$ and agitation until the precipitate is distinctly whitened by the excess of $BaCO_3$: then shake well and allow the flask to stand by corked for at least fifteen minutes, occasionally shaking it vigorously. Let the precipitate subside, filter; wash the precipitate with a little cold water allowing the washings to run through into the filtrate, then wash thoroughly, rejecting the washing-water:—

<table>
<tr>
<td colspan="2">1. *Precipitate* may contain Fe, Al, Cr, and $BaCO_3$; dissolve it in as little boiling HCl as possible; remove Ba from the *boiling* solution by adding dilute H_2SO_4 gradually, until after allowing the precipitate to settle, a few additional drops of acid cause no further precipitate (Note 1, below); filter, add pure NaHO in excess to the filtrate, boil and filter:</td>
<td colspan="3">2. *Filtrate* may contain Zn, Mn, Ni, Co, and $BaCl_2$. Remove Ba by adding to the *boiling* liquid dilute H_2SO_4 gradually, until the last few drops produce no further precipitate in the clear liquid from which the $BaSO_4$ has been allowed to settle; filter; add pure NaHO in excess to the *cold* filtrate, stir well and filter:</td>
</tr>
<tr>
<td>*Precipitate:* examine for Fe and Cr by column 2, Table III.A (436).</td>
<td>*Filtrate:* examine for Al by Column 1, Table III.A (436)</td>
<td colspan="2">*Precipitate* may contain Mn, Ni, Co; rinse off the filter into a porcelain dish with as little water as possible, add some strong HCl and boil; evaporate nearly to dryness, add a little strong solution of $Na\bar{A}$ to the liquid, pass H_2S to saturation, filter:</td>
<td rowspan="2">*Filtrate:* pass H_2S a white precipitate indicates: *Presence of Zn*.</td>
</tr>
<tr>
<td colspan="2">*Note* 1.—The separation of Ba may be neglected here, the precipitate being at once boiled with NaHO: in this case much white $BaCO_3$ will remain with the Fe_2O_3 after fusion to separate Cr; it will, however dissolve with the Fe and cause no complication.</td>
<td>*Precipitate:* examine for Ni and Co according to Table III.B, column 1 (437).</td>
<td>*Filtrate:* add AmHO in excess; a flesh-coloured precipitate shows: *Presence of Mn*.</td>
</tr>
</table>

465. TABLE IIIc$_2$.—FOR EXAMINATIO

Remove the precipitate produced by AmCl, AmHO, and .
in dilute HCl and gently stirring and shaking the liquid : tal
Note 1) :—

<table>
<tr>
<td rowspan="3"><i>Residue</i> if black may contain NiS and CoS; examine it as directed in Table III.B, column 1 (437).</td>
<td colspan="2"><i>Filtrate</i> may contain Al, Fe, Cr, Zn, Mn. Carefully
of this observation. examine the liquid by column</td>
</tr>
<tr>
<td colspan="2">1. <i>The liquid is perfectly colourless: Absence of</i>
Boil the liquid in the dish for a few minutes unt
longer smells of H_2S, then add a small crystal KC
boil down to a small bulk; cool, add NaHO in exc
well and filter :</td>
</tr>
<tr>
<td><i>Precipitate</i> may contain Fe, Mn. Dry and fuse it on platinum foil with Na_2CO_3 and KNO_3; a blue green mass shows :
<i>Presence of Mn</i> .
Boil the foil in a porcelain dish with water for some time, and if any undissolved residue is left, decant the liquid, boil the residue with HCl, and add KCyS; a blood-red coloration shows :
<i>Presence of Fe</i> (Note 3, 436).</td>
<td><i>Filtrate</i> may contain
Divide into two equ
(Note 2).
Into one portion pas
white precipitate f
once :
<i>Presence of Zn</i>
To the other portion
lute HCl graduall
stirring ; if no pre
forms, add HCl in
then AmHO in exc
heat ; a colourless
lent precipitate :
<i>Presence of Al</i></td>
</tr>
</table>

Note 1.—A mere milkiness, due to the separation of sulphur, sho
Note 2.—Al and Zn may also be detected without dividing the filtr
colourless flocculent precipitate shows *Presence of Al:* this is filtered off a

GROUPS III.A AND III.B, WHEN MIXED.

from the filter by opening the filter out inside a porcelain dish, pouring
the filter paper, stir well, and if any *black* residue is left, filter (see

<table>
<tr><td colspan="3">olour, which is best seen by pouring it into a white porcelain dish, and according to the result</td></tr>
<tr><td colspan="3">II. <i>The liquid has a violet or bright green colour : Presence of Cr.</i>
Boil the liquid in the dish until H_2S is no longer smelt, drop in a small crystal of $KClO_3$ and boil down nearly to dryness ; dilute with a little water, pour into a small flask and add $BaCO_3$ suspended in water gradually whilst constantly shaking the liquid until the excess of $BaCO_3$ whitens the precipitate, cork the flask and allow it to stand for not less than fifteen minutes, occasionally shaking it well ; filter, wash first with cold water letting the washings run into the filtrate, then with boiling water rejecting the washings :</td></tr>
<tr><td rowspan="2"><i>Precipitate</i> may contain Fe, Al, Cr ; examine it by column 1, Table III.C_1 (464).</td><td colspan="2"><i>Filtrate</i> may contain Zn, Mn ; boil, and whilst boiling add dilute H_2SO_4 gradually until the last few drops cause no further precipitate, filter from $BaSO_4$; cool, add pure NaHO in excess, stir well and filter :</td></tr>
<tr><td><i>Precipitate :</i> dry and fuse with Na_2CO_3 and KNO_3 on platinum foil ; blue green mass :
<i>Presence of Mn.</i></td><td><i>Filtrate :</i> pass H_2S ; white precipitate :
<i>Presence of Zn.</i></td></tr>
</table>

absence of Ni and Co, and does not render filtration necessary.
adding to it HCl gradually until it becomes acid, then AmHO until alkaline, a
passed into the filtrate, a white precipitate shows *Presence of Zn*.

TABLE III.D.—FOR EXAMINATIO

466. If $(PO_4)'''$ is found in the HCl solution in the Gene forms on addition of AmCl and excess of AmHO, the liquid containi is washed well with hot water. To the filtrate Am_2S is added i for Groups IV. and V. (419, 431). The two precipitates yield well with a little Am_2S, then filtered, and the residue on the fil adding $MgSO_4$; if this is present Fe, Zn, Mn, Ni, Co, one or mo

The residue left after stirring with Am_2S may contain Ba, Sr, C hydrates. Remove it from the filter and heat it with dilute HCl crystals of $KClO_3$, and evaporate very nearly to dryness. Filter as long as any precipitate is produced. Heat gently, and filter wh

<table>
<tr><td colspan="4"><i>Filtrate</i>: Add Fe_2Cl_6 (Note 2) drop by drop as long as a precipitate forms, and until th liquid, after being well stirred or shaken, remains reddish: addition of Fe_2Cl_6 i large excess must be carefully avoided. Warm gently for some time, filter whilst hot, and wash with hot water:</td></tr>
<tr><td colspan="3"><i>Filtrate</i>: Add AmCl, AmHO (Note 3), and Am_2S: filter:</td><td rowspan="3"><i>Precipitate</i> containing $FePO_4$ may be neglected.</td></tr>
<tr><td colspan="2"><i>Filtrate</i>: Add Am_2CO_3: filter:</td><td rowspan="2"><i>Precipitate</i>: examine by Table III.C. (464, 465) for Zn. Mn, Ni, Co, also for Al and Cr.
Test a portion of the original solution, or the solution of the substance in HCl, for Fe'' and Fe''' by note 3 (436).</td></tr>
<tr><td><i>Precipitate</i> may contain $BaCO_3$, $SrCO_3$, $CaCO_3$. Examine this precipitate by Table IV. (438). Ba, Sr, Ca, if found in this precipitate, were present in the solution as phosphates.</td><td><i>Filtrate</i> may contain Mg; add Na_2HPO_4, warm and shake well; white crystalline precipitate: <i>Presence of Mg as phosphate.</i></td></tr>
</table>

Note 1.—An insoluble residue here may contain $SrSO_4$ and $BaSO_4$ and mu sent originally as phosphates.

Note 2.—A few drops of Fe_2Cl_6 may be added to a small part only of t at once for Groups III. and IV. and for Mg, without adding Fe_2Cl_6.

Note 3.—Since this filtrate has to be tested for Groups III.A and III.B, analysing the precipitate by Table III.C, or by precipitating Groups III.A a cipitates by Tables III.A and III.B (436, 437).

Note 4.—Since $CrPO_4$ is rarely present, this precipitate may generally be teste detected by acidifying with HCl and adding AmHO in excess which gives ing in HCl and adding KCyS.

OF PHOSPHATES IN GROUP III.

Table (419) after precipitating Groups I. and II., and a precipitate
the precipitate is gently heated, filtered quickly, and the precipitate
excess and the solution boiled, then filtered, and the filtrate examined
by AmHO and by Am_2S are transferred to a porcelain dish and stirred
is washed well. The filtrate should be examined for $(PO_4)'''$ by
were present as phosphates.

Mg as phosphates; Fe, Zn, Mn, Ni, Co as sulphides; and Al, Cr as
a porcelain dish. If this does not dissolve it, drop in several small
S if necessary (Note 1); then add a solution of $H\bar{A}$ and $Na\bar{A}$ (526. 84),
hot:—

<table>
<tr><td colspan="4"><i>Precipitate</i> may consist of $FePO_4$ $AlPO_4$, $CrPO_4$ (Note 4). Dry the precipitate on the filter, and fuse it for a short time in a platinum crucible or on platinum foil with a mixture of finely powdered SiO_2, Na_2CO_3, and KNO_3. When cold dissolve by boiling with a little distilled water, add Am_2CO_3; allow to stand for a short time, stirring occasionally; let the precipitate subside, filter:</td></tr>
<tr><td rowspan="3"><i>Filtrate:</i> add $H\bar{A}$ until the solution is acid, boil for a short time, then add $Pb\bar{A}_2$; yellow precipitate of $PbCrO_4$: <i>Presence of Cr, as phosphate.</i>
<i>Note.</i>—A white precipitate on addition of $Pb\bar{A}_2$ may be disregarded.</td><td colspan="3"><i>Precipitate</i> may contain H_4SiO_4, Fe and Al as silicates, and Fe_2Ho_6. Acidify with HCl, evaporate to dryness, and heat the dry residue gently. Warm with a few drops of strong HCl, add hot water, and filter:</td></tr>
<tr><td colspan="2"><i>Filtrate:</i> add pure NaHO in excess, boil and filter:</td><td rowspan="2"><i>Residue</i> is SiO_2 and may be neglected.</td></tr>
<tr><td><i>Filtrate:</i> acidify with HCl, and add AmHO in excess, boil: white, gelatinous precipitate: <i>Presence of Al as phosphate.</i></td><td><i>Precipitate</i> is brown Fe_2Ho_6: dissolve by boiling with HCl, and add KCyS: blood-red coloration: <i>Presence of Fe as phosphate.</i></td></tr>
</table>

be examined by the latter part of (428): Ba or Sr, if found here, were pre-
filtrate and heated; if it causes no precipitate the other portion may be examined

may be examined either by adding AmCl, AmHO, and Am_2S together and
III.B separately by AmCl and AmHO and then Am_2S, and by examining the pre-

by boiling with excess of NaHO, and filtering; $AlPO_4$ will be in solution, and is
gelatinous precipitate; Fe_2Ho_6 remains as a precipitate and is detected by dissolv-

467. ANALYSIS OF METALS AND ALLOYS.

After making the Preliminary Examination given below either of the two methods (470 or 471) may be adopted. The first method is by far the most ready and certain for the examination of a metal or alloy of unknown composition, since all metals, even when present in small quantity only, may thus be detected.

In making the solution by the second method, Sn, Sb, Au, and Pt are left undissolved, all other metals passing into solution; it is not however to be recommended as a *general* process for examination of metals and alloys, since the following complications are apt to occur:—If As is present with Sn, a part or the whole of the As may remain in the residue; Pt, if present with a sufficient quantity of Ag, may pass entirely into solution; and Sb will always partly dissolve. The process however is useful in cases where an alloy, known to contain a large proportion of Sn or Sb, has to be examined for other metals, since these are dissolved away at once from the bulk of the Sn or Sb: but care must be taken that As is not passed over in the presence of Sn.

PRELIMINARY EXAMINATION.

Note the colour, and also if any smell is given off when the substance is rubbed with the hand; also whether it is crystalline or not, and if it is attracted by a magnet (Fe, Ni, Co, &c.). Note also the hardness of the metal, by seeing if it can be scratched or cut by a steel knife; also whether on being struck smartly with a hammer it breaks to powder (brittle), or flattens out (malleable): then try the following experiments:—

Experiment.	Observation.	Inference.
468. I.—Heat a small portion on charcoal in the inner blowpipe flame.	The results obtained by (403) and (404) in the Preliminary Table may be here noticed.	The inference will b found stated in (403 and (404).
469. II.—Heat a portion in a small tube closed at one end.	A metallic sublimate forms.	Presence of Hg, Cd, As the two latter are detected by (468).
Note.—As and S can frequently only be detected as arsenate and sulphate by fusion with KNO_3 and Na_2CO_3, dissolving in water, and testing the solution by (449) or (446).	The sublimate consists of small liquid globules.	Presence of Hg.
	A yellow sublimate melting to reddish-yellow drops; the substance when heated *slowly* in a tube open at both ends evolves SO_2 (417).	Presence of S from a sulphide.

PROCESSES FOR SOLUTION AND EXAMINATION OF A METAL OR ALLOY.

470. METHOD I.—Pour some rather dilute HCl upon the powdered metal in a small flask covered with a watch-glass with its concave surface upwards, and heat for some time just short of boiling; if the metal dissolves readily, continue heating until the metal is completely dissolved, and examine the HCl solution according to the General Table (419).

Frequently HCl alone does not effect complete solution, two or three drops of strong HNO_3 should then be poured in, and more HNO_3 and HCl added occasionally when the action ceases or when red fumes are no longer given off on heating. When the metal has entirely disappeared, add a little more strong HCl and boil as long as any Cl or reddish fumes are given off; then dilute with a little water, heat to boiling, and cool; filter if there is any white residue :—

Residue, if crystalline, is probably $PbCl_2$, and will be found to dissolve entirely if washed with sufficient boiling water. In this solution the presence of Pb is confirmed by adding K_2CrO_4, which gives a yellow precipitate soluble in excess of KHO, showing *Presence of Pb.* If any residue is left after washing well with boiling water, it is probably AgCl ; confirm the presence of Ag by pouring upon the residue hot AmHO, it dissolves completely but is reprecipitated on adding HNO_3 in excess :— *Presence of Ag.*	*Filtrate* (note 1): dilute largely with H_2O (see note 2), and whether this causes a precipitate or not, pass H_2S to saturation into the solution ; examine any precipitate thus produced by Table II. (435), and proceed to examine the filtrate for Groups III., IV., and V., as directed in the General Table (419). *Note* 1.—If Au and Pt may be present, they must be tested for in the H_2S precipitate, according to the directions in (472–474). *Note* 2.—A white precipitate appearing on dilution is due to the presence of Bi, Sb, or Sn.

471. METHOD II.—Pour upon the finely divided metal some strong HNO_3*, and heat in a small flask, covered with a watch glass, as long as any red fumes appear: one of two results will occur:—

---

* If Hg is found in the preliminary examination and small quantities of Ag have to be tested for, the Hg should be expelled by heating the substance strongly in a porcelain crucible before dissolving in acid, as $Hg(NO_3)_2$ tends to prevent the precipitation of Ag by HCl in Group I.

I.	II.	
The substance dissolves completely with or without addition of water. Absence of Pt, Au, Sb, Sn (note 1). Examine the solution, after boiling nearly to dryness and diluting with water (note 2), by the General Table (419).	*A residue is left:* add some hot water and boil, then filter and wash the residue on the filter well with boiling water (note 2). The residue may present the following appearances:—	
	1. *Entirely metallic or black powder:* probably Pt or Au. Dissolve by heating in a small flask covered with a watch glass, with a little HCl to which a few drops of HNO_3 have been added. When completely dissolved add more HCl, and boil down in an evaporating basin nearly to dryness, examine the solution for Au and Pt by (474), commencing with the addition of KCl and using only the left hand side of the table, since Sn will be absent.	2. *White powder* may contain Sn, Sb, As, possibly also Pt and Au concealed in it (note 3).

Note 1.—The solution is liable to contain small quantities of these metals, which must always be tested for in a careful analysis.

Note 2.—BiOCl will often precipitate on dilution, but the precipitate will disappear on adding HCl and boiling, being thus easily distinguished from the Group I. precipitate.

Note 3.—If much residue is obtained a small quantity of it may be heated in a test-tube with HCl adding $KClO_3$, if it dissolves entirely dissolve the whole of the residue, then examine the solution by Table II., commencing at (435 *b*): if it refuses to dissolve proceed with the rest of the residue as directed below. If the residue is small in quantity examine it at once as directed below.

Examination of the Non-metallic Residue insoluble in HNO_3.—Dry the residue on the filter at a gentle heat, mix it thoroughly with about an equal quantity of powdered Na_2CO_3 and $NaNO_3$, and add the mixture gradually to some $NaNO_3$ in fusion in a porcelain crucible; then pour the melted substance out into a porcelain dish; allow it to cool, then pour upon it cold water and let it stand for some time. After crushing the mass with a pestle and stirring it occasionally, filter, and wash the residue on the filter with dilute alcohol, throwing away the washings:—

Residue may contain Sn, Sb, Pt, Au. Place it in a small porcelain dish, pour in a little HCl and heat, then add water; whether the residue has dissolved or not place in the liquid a strip of Pt-foil and drop upon it a piece of pure Zn; H is given off; wait until this ceases, taking care that there is some Zn left undissolved, and adding more Zn if the first piece has entirely dissolved. Now remove the platinum strip; if it is stained black *Sb is present.* The residue in the dish may consist of Sn, Au, and Pt; remove the Zn, rinsing off any substance adhering to it into the dish, stir the liquid in the dish well, then pour off the liquid, carefully leaving the residue; pour in water, stir well, and again pour off carefully. Boil the residue for some time with strong HCl in a test-tube, dilute, decant, add $HgCl_2$, a white precipitate forms :—*Presence of Sn.* *Residue:* dissolve by warming with HCl and HNO_3, and examine the solution for Au and Pt by (474), using only the left hand portion of the table.	*Filtrate* may contain $(AsO_4)'''$: add HNO_3 until the solution is acid and boil, evaporating the liquid in a dish if very bulky. Pour into half this solution $AgNO_3$ as long as it gives any precipitate, and add gradually AmHO diluted with 10 or 12 times its bulk of water, a brown precipitate shows :— *Presence of As.* To the other half of the acid solution add AmHO in excess; then $MgSO_4$, and rub the inside of the vessel with a glass rod; a white crystalline precipitate, often appearing only after some time, shows :— *Presence of As.*

SEPARATION AND DETECTION OF Au AND Pt.

472. Au and Pt will, in the ordinary course of analysis, be entirely precipitated as sulphides in the second Group, if H_2S is passed for some time into the hot HCl solution; and since these sulphides are soluble in KHO and in Am_2S, on examining the H_2S precipitate by Table II. (435), the Au and Pt will pass into the filtrate when the precipitate is boiled with KHO or Am_2S. No modification of the process described in Table II. is required for the detection of these two metals until the examination of the residue in the hydrogen-flask is commenced. The Au and Pt will be present in this residue, associated with Sn if it be present, and usually also with at least a part of the Sb (if present), since Zn and Pt in contact in an acid liquid cause the separation of metallic Sb (193). Accordingly when Au and Pt have to be tested for, the ordinary course of analysis is to be followed until the residue in the hydrogen-flask is obtained; and this is examined as directed in (473).

473. After washing the residue left in the hydrogen-flask by decantation in a porcelain dish, and removing any excess of Zn, boil it with a little strong HCl in a test-tube for several minutes, allow the residue to settle, and decant the liquid :—

<table>
<tr>
<td colspan="2">474. <i>Residue</i>*: pour upon the residue in the dish a little HCl, add several drops of HNO_3, and boil gently very nearly to dryness; add some KCl solution and evaporate once more very nearly to dryness. Pour some absolute alcohol into the cool dish and stir well for a time, allow the precipitate to settle and decant the liquid, wash the precipitate by stirring it with a little more alcohol and decant the liquid when the precipitate has settled :—</td>
<td rowspan="2"><i>Solution</i>: to one part add $HgCl_2$ a white precipitate, which does not appear at once if only traces of Sn are present:
<i>Presence of Sn</i>.
Pour the other part of this solution into a porcelain dish, immerse in it a strip of Pt and drop a piece of Zn on the Pt; a black stain on the Pt shows:—
<i>Presence of Sb</i>.</td>
</tr>
<tr>
<td><i>Precipitate</i> will consist of yellow K_2PtCl_6 and excess of KCl, dissolve it in a little boiling water, pour it into a white dish, add several drops of HCl, then $SnCl_2$: an orange-red coloration confirms the
<i>Presence of Pt</i>.</td>
<td><i>Solution</i> will be yellow if Au is present; evaporate carefully on a water-bath just to dryness, dissolve in a little water and add a few drops of freshly prepared $FeSO_4$ solution. Au will be precipitated in a fine powder, causing the liquid to appear reddish by reflected and blue by transmitted light:—
<i>Presence of Au</i>.</td>
</tr>
</table>

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* If Sb has not been detected already in the precipitate produced by the gases in $AgNO_3$ solution (435 *b*) this residue should be examined for Sb, as the Sb instead of having been given off as SbH_3, may have been deposited on the Pt or Au. Boil it once more for some time with strong HCl to remove all Sn, wash the residue well by decantation and boil it in the dish with $H_2\bar{T}$ adding a few drops of HNO_3, decant, and test the liquid for Sb by adding HCl and passing H_2S : the residue is then examined as above (474).

EXAMINATION OF SUBSTANCES INSOLUBLE IN WATER AND ACIDS.

475. *An insoluble substance may consist of* one or more of the following substances, all of which are white except Fe_2O_3, Cr_2O_3, $FeCr_2O_4$, $PbCrO_4$, S, C, and AgCl which has been exposed to light.

In the following list those substances which are embraced in brackets ( ) may *possibly* be present, those in square brackets [ ] *improbably* because they are soluble in water.

1. $BaSO_4$	Insoluble in water and acids.
2. $SrSO_4$	Insoluble ,, ,,
3. $[CaSO_4]$	Not perfectly insoluble in water, soluble in hot HCl: hence it should pass into the acid solution.
4. $(PbSO_4)$	
5. $PbCrO_4$	Insoluble after being strongly heated.
6. $[PbCl_2]$	Soluble in boiling water, and should therefore have been removed if the residue was well washed with boiling water.
7. $AgCl$	This may have been originally present as such, or may have been derived from the use of HCl in making the solution, or by the action of aqua regia on the insoluble substances AgBr, AgI, AgCy, $Ag_6Fe_2Cy_{12}$, Ag_4FeCy_6.
8. SiO_2	Either uncombined, or as a silicate.
9. (Al_2O_3)	Insoluble after being strongly ignited, but these are usually dissolved by long boiling with strong HCl.
10. (Fe_2O_3)	
11. $(Cr_2O_3$	
12. $FeCr_2O_4$	Chrome iron ore, native.
13. (SnO_2)	Native or ignited.
14. Sb_2O_5, Sb_2O_4	
15. CaF_2	Also a few other fluorides, and some metaphosphates and arsenates.
16. S	Yellow, slowly soluble in strong HNO_3, giving red fumes, and yielding H_2SO_4.
17. C	Black, and quite insoluble.

If sufficient of the substance is at disposal, the preliminary examination (476) may be made on a portion of it. In case the quantity of substance is small, however, the whole of the substance must be employed for the examination by fusion (477).

476. Preliminary Examination.

The substance must be in the state of dry powder. Make a careful examination of it with a pocket lens. Expts. I. and II. need only be made if the substance is light in colour.

Experiment.	Observation.	Inference.
I. Observe whether the substance darkens when allowed to stand in the light for some time.	The colour changes to violet or black.	Presence of AgCl.
II. Pour a little Am_2S upon a portion of the substance on a watch glass.	The substance blackens; pass on to III. The substance does not blacken; pass on to V., omitting III. and IV., since Pb and Ag must be absent.	Presence of Pb or Ag.
III. Heat some of the substance with a little water and a small piece of KCy; filter off, keeping the residue; to the filtrate add Am_2S.	A brownish precipitate.	Presence of AgCl. *Conf^y.* On warming some of the substance with AmHO, filtering, and adding excess of HNO_3 to the filtrate, a white precipitate forms, which, when shaken well or heated, coagulates into flocks.
IV. Wash the residue from Exp. III. well upon the filter:—		
***a. It is white;* drop Am_2S upon it.**	The residue blackens.	Presence of $PbSO_4$ or $PbCl_2$.
***b. It is dark coloured;* pour in a little $H\bar{A}$, and boil after adding AmHO in excess; filter, to the filtrate add $H\bar{A}$ in excess and K_2CrO_4.**	A yellow precipitate, soluble in KHO.	Presence of $PbSO_4$ or $PbCl_2$.
V. Take up some of the substance upon a moistened loop of platinum wire; heat it for a short time in the inner blowpipe flame, then moisten with a drop of strong HCl, and hold in the outer part of a Bunsen flame.	A *reddish-yellow* coloration, *dusky-green* when seen through the indigo-prism. A *crimson-red* coloration appearing *deep-red* through the indigo-prism. A *yellowish-green* coloration. *Note.*—If all these are present, the colorations may often be seen in succession.	Presence of Ca Presence of Sr Presence of Ba as sulphate.
VI. Heat in a small test-tube or ignition-tube, then strongly on a piece of porcelain or broken glass, or on platinum foil.	A yellow sublimate forms on the sides of tube. When strongly heated the substance smoulders, and ultimately burns away.	Presence of S. Presence of C.

Experiment.	Observation.	Inference.
VII. Heat with strong H_2SO_4 in a platinum crucible or leaden cup covered with a watch glass (296): or if a silicate is present examine by passing the gas into AmHO (297).	The glass is etched. A gelatinous precipitate is obtained in the AmHO.	Presence of F. Presence of F.
VIII. Fuse some of the substance in a bead of $NaAmHPO_4$, first in the outer then in the inner blowpipe flame.	Particles are seen floating undissolved in the melted bead. *Green-coloured* bead. *Reddish-brown* bead, colourless when cold, and becoming *greenish* in the inner flame.	Presence of SiO_2 (480). Presence of Cr. Presence of Fe.

Examination of Insoluble Substances.

477. Mix the finely powdered substance* with three or four times its weight of fusion-mixture, and heat the mixture in a small covered porcelain crucible† until it melts; keep it in fusion for at least ten minutes. Allow the crucible to cool, then pour some water into it and allow to stand or boil until the solid mass is loosened from the crucible; boil this in a porcelain dish with distilled water, crushing the mass by pressure with a pestle if it does not quickly fall to pieces. Allow the residue to settle, pour off the solution through a filter, and boil the residue with a little more water; pour off through the same filter, adding this filtrate to the former one.

For the examination of the undissolved residue see (478), for the filtrate (479).

478. *Residue on the filter;* wash well with boiling water, then make a hole in the bottom of the filter, wash the residue through into a test-tube by pouring upon it a little boiling dilute HNO_3 (see note 1 below), and boil; the residue dissolves entirely if the fusion has been continued sufficiently long (see note 2). Filter if necessary, and examine the solution by the General Table (419), bearing in mind that only the metals enumerated in (475) are likely to be present (note 3).

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* Which, if it is found by Exp. VI. to contain free S or C, must first be heated strongly for some time in an open porcelain crucible.

† If Pb and Ag have been proved to be absent by the preliminary tests, or if they have been first removed by boiling the substance with KCy solution, then with $H\bar{A}$ and excess of AmHO and washing well, a platinum crucible may be used. The use of a platinum crucible is preferable, since after fusing in porcelain small quantities of Al_2O_3 and SiO_2 will always be found, being derived from the action of the Na_2CO_3 on the glaze of the porcelain.

Note 1.—If Ag and Pb are known to be absent by the preliminary experiments, HCl may be used for dissolving the residue and is preferable; if effervescence is produced by these acids the presence of Ba, Sr, Ca or Mg is proved.

Note 2.—A residue here may consist of chrome iron ore; this may be dissolved by heating with HNO_3 and $KClO_3$, and the solution tested by the General Table, when Cr and Fe will be found.

Note 3.—It is best to evaporate the solution quite to dryness before testing for Group III., using a water-bath towards the end if the substance spirts. This is always necessary if SiO_2 is present in the substance, since it is sometimes obtained in the acid solution, and is by this means at once separated.

479. *Aqueous solution of the Fused mass.*—Divide into two parts A and B.

A. Add HCl until the liquid is distinctly acid, and evaporate in a porcelain dish to dryness, finishing the process on a water-bath if necessary to avoid spirting; then continue to heat the dish gently until the residue is quite dry; pour in a little strong HCl and warm, dilute and heat again; an insoluble residue shows *presence of* SiO_2.

Filter, and examine the filtrate by the General Table (419) more particularly for Al, but other bases should also be tested for, which being soluble in alkalis may pass into this solution, such as Cr, Mn, Zn, Sn.

B. Test separate portions for the following acid-radicles as described below:—

1. *Chloride:* acidify with HNO_3 and add $AgNO_3$, white precipitate easily soluble in AmHO.
2. *Sulphate:* acidify with HCl and add $BaCl_2$, white precipitate insoluble on boiling.

Note.—Unless the fusion mixture employed was free from chloride and sulphate, tests 1 or 2 are worthless.

3. *Chromate:* acidify with $H\bar{A}$ and add $Pb\bar{A}_2$, yellow precipitate; a chromate is seen also by the yellow colour of the solution. Cr thus detected may have been present acting as a metal or as a constituent of an acid-radicle.
4. *Phosphate:* acidify with HNO_3, add a few drops to $AmHMoO_4$ solution, and warm gently. A yellow precipitate forms, often only after a time, or on gently heating. If As has been found on passing H_2S into the hot HCl solution, $(PO_4)'''$ must be tested for in a portion of the filtrate after boiling off H_2S, else the above yellow precipitate may be due to $(AsO_4)'''$.
5. *Fluoride:* add HCl in excess to a part of the solution, stir well, and let stand until the CO_2 has escaped, then add AmHO in excess, then $CaCl_2$ as long as it produces any precipitate, and let stand for a time; filter off, dry the precipitate and examine it for F by pouring strong H_2SO_4 upon it in a platinum crucible covered with a watch glass (296).

ANALYSIS OF SILICATES.

480. The presence of silica is shown by Exp. VIII. in the Preliminary Examination of insoluble substances (476); when it has been found it becomes necessary to examine for all metals, since many silicates which are soluble when alone, become insoluble when mixed or combined with insoluble silicates.

The ordinary analytical course requires but slight modification to adapt it to the examination of silicates. Many silicates are entirely decomposed by heating with strong HCl for some time just short of boiling; if this decomposition has been effected, only a colourless residue of silicic acid will remain, which may be identified by its solubility in hot Na_2CO_3 solution.

If the silicate is not completely decomposed by hot strong HCl, some of it must be fused with fusion mixture as directed in (477), and the cool residue boiled with water; the solution and residue are then examined by (478) and (479), remembering however that all metals may be present.

481. Since Na and K cannot be tested for in the solution obtained after fusion, a separate portion must be examined for K and Na by one of the following processes:—

I. Evaporate the powdered substance several times in a platinum dish or crucible either with hydrofluoric acid and subsequently with strong H_2SO_4, or with five times its weight of calcium fluoride mixed into a paste with strong H_2SO_4, in either case heating finally until no more white fumes are evolved. Boil the cool residue with water, add $BaCl_2$ solution as long as it causes any precipitate, then add AmHO in excess and Am_2CO_3 as long as it causes any precipitate, filter and examine the filtrate for K and Na by Table V. (439).

II. Mix the powdered substance intimately with four times its weight of barium hydrate, and heat strongly in a platinum crucible for about half an hour; dissolve the cool mass in dilute HCl, add AmHO in excess and then Am_2CO_3 in excess, filter, rejecting the precipitate; evaporate the filtrate to dryness, ignite the residue, then boil it with water; add a little HCl to the solution and test for K and Na by Table V. (439).

482. ANALYSIS OF SUBSTANCES CONTAINING CYANOGEN.

If a small quantity of the substance to be analysed, when examined by (304) or (306), is found to contain cyanogen, the usual course of analysis must be somewhat modified, since the presence of cyanogen would produce confusing results. The cyanogen may be present as a cyanide, sulphocyanide, ferrocyanide, ferricyanide, or cobalticyanide, rarely as a manganocyanide or chromicyanide. Since the method to be adopted when a cyanide or sulphocyanide only is present, is much more simple than that rendered necessary by the presence of the other cyanogen-compounds above mentioned, it is advisable first to ascertain in what form the cyanogen occurs, by the following preliminary experiments made on a small portion of the substance; according to the results yielded by these experiments the further examination of the substance is made by Method I. (484) or by Method II. (485).

PRELIMINARY EXAMINATION.

483. Boil a portion of the substance for several minutes with KHO solution, then add some Na_2CO_3 solution as long as it causes any precipitate, and boil again for several minutes; filter, make the cold filtrate just acid with HCl, filter if necessary, and test separate portions as follows :—

Reagent added.	Result.	Inference.
1. $FeSO_4$ solution . .	Blue precipitate .	Presence of $(FeCy_6)^{iv}$ $(FeCy_6)_2^{vi}$.
	White precipitate .	Probable presence of $(CoCy_6)_2^{vi}$.
2. Fe_2Cl_6 solution . . .	Blue precipitate .	Presence of $(FeCy_6)^{iv}$.
	Blood-red coloration	Presence of $(CyS)'$.
3. $ZnSO_4$ solution as long as it causes any precipitate.	Light-brown precipitate . . .	Presence of $(FeCy_6)_2^{vi}$.
	White precipitate .	Presence of $(FeCy_6)^{iv}$, $(CoCy_6)_2^{vi}$.
Confirmatory.—Filter off the precipitate, add $ZnSO_4$ solution to the filtrate, and if it causes any precipitate, pour again through the filter, repeating this process until no further precipitate is produced by $ZnSO_4$. Fuse some of the precipitate in a clear borax bead	A blue bead is produced	Presence of $(CoCy_6)_2^{vi}$.

If these preliminary tests show the presence of (FeCy$_6$)^{iv}, (FeCy$_6$)$_2^{vi}$ by Method II. (485). If the absence of these acid-radicles is indi- indicate their presence, proceed according to Method I. (484).

If none of the above acid-radicles are found in the preliminary course is to expel HCy by boiling after adding HCl in preparing

484. METHOD I.—If (CyS)' is present, boil the portion of the for some time, dilute a little and boil again. If any residue remains as directed at the end of (420), any undissolved residue being evaporated nearly to dryness and examined by (419).

Another portion of the substance is examined for acid-radicles in

485. METHOD II. (Fresenius).—Boil the substance with water, to the filtrate :—

<table>
<tr>
<td rowspan="5"><i>Filtrate</i> : examine this for metals, more especially the alkalis, and for acid-radicles according to the directions given for analysing a liquid (350).
The acid-radicles should be first tested for in a portion of the solution and if $(FeCy_6)^{iv}$, $(Fe_2Cy_{12})^{vi}$, $(Co_2Cy_{12})^{vi}$, one or more be found, the solution must be evaporated to dryness with HNO_3, and the residue strongly heated (see General Table) after filtering from the H_2S precipitate, in order to destroy these cyanogen radicles. The Fe, Co, &c., in the cyanogen acid-radicles will then be detected in the General Table.</td>
<td colspan="3"><i>Residue</i> : boil with KHO solution for several minutes, then add and boil again ; filter and wash the residue :—</td>
</tr>
<tr>
<td colspan="3"><i>Filtrate</i> : pass H_2S, and if it causes any precipitate continue KHO[*], heat and filter :—</td>
</tr>
<tr>
<td colspan="2" rowspan="2"><i>Precipitate</i> : wash well with boiling water, and boil the precipitate with strong HNO_3, a black residue of HgS may remain, filter this off after diluting the acid. and confirm the presence of Hg in the precipitate by heating it with Na_2CO_3 in a bulb-tube (147).
The filtrate (or solution if HgS is absent) is evaporated to dryness, the residue is then dissolved in a little hot strong HCl, the solution is much diluted, saturated with H_2S, and filtered :—</td>
<td><i>Filtrate</i> : add dilute saturation and filter :—</td>
</tr>
<tr>
<td rowspan="2"><i>Precipitate</i> : boil with KHO, filter off any black precipitate and examine it for Hg by heating it with Na_2CO_3 in a bulb-tube (147).
Add to the filtrate or solution HCl until it is acid, pass H_2S to saturation, and examine for As, Sb, Sn, (Pt, Au), by Table II. (435), commencing at (435 <i>b</i>).</td>
</tr>
<tr>
<td><i>Precipitate</i> : examine for Pb and Cu by Table II. (435 <i>a</i>).</td>
<td><i>Filtrate</i> : add AmCl, AmHO in excess, and Am_2S, and examine for Zn, Mn, Ni, Co, &c., by Table III.C_2 (465).</td>
</tr>
</table>

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[*] If H_2S causes a precipitate to form, NaHS or KHS may be added, the filtrate ; this may be done instead of passing H_2S to saturation and then

$(CoCy_6)_2^{vi}$, one or more, examine the remainder of the substance
cated by not obtaining the results in the second column which

experiments, the only modification requisite in the ordinary analytical
the solution (383) or in testing for Group I. (419).
substance which is to be examined for metals with strong HNO_3
decant the solution and proceed to endeavour to dissolve the residue
then examined according to (475 *et seq.*). The HNO_3 solution is

the usual way.
filter, and wash the residue with boiling water, adding the washings

<table>
<tr><td colspan="4">some Na_2CO_3 solution as long as it causes any precipitate in the solution or the filtrate,</td></tr>
<tr><td colspan="3">to pass the gas until the liquid is saturated, then add more</td><td rowspan="5">Residue: dissolve and examine for metals in the usual manner (383).
Fe, Co, Mn, Cr, if found in this residue, were not present as $(FeCy_6)^{iv}$, $(Fe_2Cy_{12})^{vi}$, $(Co_2Cy_{12})^{vi}$, &c.</td></tr>
<tr><td colspan="3">HNO_3, gradually until the liquid is just acid, pass H_2S to</td></tr>
<tr><td colspan="3">Filtrate: Divide into two parts, α, β:—</td></tr>
<tr><td rowspan="2">α. Examine for acid radicles in the usual way, testing for $(Co_2Cy_{12})^{vi}$ by adding excess of $ZnSO_4$, filtering and trying whether the precipitate gives a blue bead with borax.</td><td colspan="2">β. Evaporate to dryness and fuse the residue: when cold boil it with water and filter:—</td></tr>
<tr><td>Residue: dissolve in HCl, and test for Al, Fe, Mn, Co (465); the last three if found were present as cyanogen acid-radicles.</td><td>Filtrate: acidify a portion if yellow, with $H\bar{A}$ and add $Pb\bar{A}_2$, a yellow precipitate shows $(CrO_4)''$, the Cr having been present as cyanogen acid-radicle.
Test another part for Al by adding HCl in excess, then AmHO in excess.</td></tr>
</table>

drop by drop, until it no longer gives any precipitate in the liquid or in
adding KHO.

486. The advantage of employing this somewhat complicated and tedious method of Fresenius is that it usually enables the analyst to ascertain whether Fe, Co, Mn, Cr, when present, form a part of a complex cyanogen acid-radicle or simply act as metals combined with acid-radicles. For if Fe, Co, Mn, Cr, are found in the residue left after boiling with KHO and Na_2CO_3, they were present in the latter form, if found in the alkaline filtrate they were present as cyanogen acid-radicles; if they were detected as cyanogen acid-radicles in the aqueous solution they were entirely present in that solution in that form, although they will also be detected in the examination for metallic-radicles; if they were detected only as metallic-radicles and not as cyanogen acid-radicles, they were present as metallic-radicles in the aqueous solution.

487. The following two processes have been proposed for removing cyanogen from a substance before analysis, after which the substance may be examined for metals in the usual way (382, *et seq.*); but these methods, though much more easy and rapid, are less perfect than the preceding ones, since Fe, Co, Mn, Cr, if detected, may have been present either as cyanogen acid-radicles, or as metals united with an acid-radicle, or in both forms.

1. Pour upon the powdered substance strong H_2SO_4 in a porcelain crucible, evaporate to dryness and ignite the residue strongly; when cold dissolve it by heating with a little strong HCl, adding water and heating again. (Rose.)

2. "Cyanogen compounds are best destroyed by fusion in a porcelain crucible with 3 or 4 times their weight of a mixture of 3 parts of Am_2SO_4 and 1 part of $AmNO_3$." The residue left in the crucible is dissolved and examined for metals in the usual way.

EXAMPLE SHOWING HOW TO ENTER THE RESULTS OF ANALYSIS OF A COMPLEX SUBSTANCE.

The substance given for analysis consisted of a powder containing pink, blue, white, and black particles. It smelt faintly of ammonia.

488. PRELIMINARY EXAMINATION FOR METALS.

Expt.	Observation.	Inference.
EXPT. I.—Heated in a small dry test-tube.	Water given off which turned red litmus-paper blue.	Pres. of H_2O. Pres. of NH_4.
	Strong smell of NH_3 gas.	Pres. of NH_4.
	Substance blackened, no smell of burning.	Prob. pres of Co, Cu, and abs. of $\bar{T}$ and $\bar{A}$.
	Slight white sublimate.	Pres. of As, NH_4, Hg.
Confy.—Held in the upper part of the tube a glass rod with a drop of lime water hanging on its end.	The lime water became milky.	CO_2 evolved.
	Brown nitrous fumes evolved, recognised by their smell.	Pres. of nitrate.
	Cl-gas evolved, found by smell and bleaching litmus.	Pres. of Cl.
Confy.—Boiled a portion of the substance with KHO solution.	NH_3 gas was evolved, recognised by its smell, and by giving white fumes with strong HCl.	Pres. of NH_4.
Confy.—Heated strongly another portion of the dried substance with Na_2CO_3 in a bulb-tube.	No mirror formed.	Abs. of Hg and As.
EXP. II.—Heated a portion of the substance on a loop of platinum wire in the Bunsen flame, moistened with HCl and heated again in the flame.	*Bright-yellow* flame.	Pres. of Na.
	The flame appeared *crimson* through the indigo-prism.	Pres. of K.
Heated for some time in the tip of the inner blowpipe flame, moistened with HCl, and again held in the Bunsen flame.	*Crimson* col. appearing *intense red* through the indigo-prism.	Pres. of Sr.
	Bright green col. with *blue* core.	Pres. of Cu.

Expt.	Observation.	Inference.
Expt. III.—Heated a portion of the substance in charcoal in the inner blowpipe flame.	The greater part of the substance fused readily, and was absorbed by the charcoal.	Pres. of a salt of K, Na.
	Deflagration occurred.	Pres. of chlorate, nitrate.
	A red metallic residue remained.	Pres. of Cu.
Confy.—Fused in a clear borax-bead in the outer and inner blowpipe flames.	In the outer flame a bead green whilst hot, blue when cold. In the inner flame red and nearly opaque.	Pres. of Cu.
Confy.—Fused on platinum foil with Na_2CO_3 and KNO_3.	No bluish-green or yellow mass on cooling.	Abs. of Mn and Cr.
Confy.—Fused on charcoal in the inner blowpipe flame with Na_2CO_3.	Red metallic residue.	Pres. of Cu.
	A portion placed on a silver coin and moistened gave no black stain.	Abs. of S.

489. Preliminary Examination for Acid-radicles.

Expt.	Observation.	Inference.
Expt. I.—Added dilute HCl without heating.	A colourless gas was evolved, which was free from smell, and turned a drop of lime water milky.	Pres. of carbonate. Abs. of sulphite, hypochlorite, &c.
Heated to boiling.	Cl was evolved, recognised by its smell and by bleaching moist litmus-paper.	Pres. of nitrate, chlorate, or some other oxidising substance.
Expt. II.—Added strong H_2SO_4.	A bright yellow chlorous gas evolved, which crackled when warmed.	Pres. of chlorate.
Confy. — Dropped in copper turnings and heated.	Reddish-brown fumes evolved.	Pres. of nitrate.
Heated strongly, cooled, and rinsed out.	The tube when dry was seen not to be etched.	Abs. of fluoride.
Confy.—Boiled a portion of the substance with water, added strong H_2SO_4, cooled and poured in $FeSO_4$ solution carefully.	A brown ring formed on the surface of the acid.	Pres. of nitrate.

490. EXAMINATION FOR METALS IN THE WET WAY.

Boiled a portion of the substance with Fe_2Cl_6, $FeSO_4$ and KHO, added HCl in excess, no blue pp. :—*Absence of Cy.*

Proceeded to dissolve the substance and examine the solution by the General Table.

(*Continued on page* 276.)

Boiled a portion of the substance with water; as it did not upon the undissolved residue: effervesence occurred, and on boilin Cl was perceived; the substance was completely dissolved. Co previously to give no pp. on addition of a little dilute HNO_3 :—

<table>
<tr><td rowspan="3"><i>No pp.</i>
<i>Abs. of Group I.</i>
Hg', Ag, and prob. Pb.</td><td colspan="2">Diluted with water and passed H_2S until the liquid smelt</td></tr>
<tr><td rowspan="2"><i>A black pp.</i>
Exam$^{d.}$ by Table II.</td><td><i>Filtrate</i> which gave no further
Boiled until it no longer smelt
on addition of HNO_3, (<i>prob.</i>
of the HCl sol$^{n.}$ to some Am
boiled; filtered :—</td></tr>
<tr><td><i>A brown pp.</i>
Exam$^{d.}$ by Table III.A.]</td></tr>
</table>

491. Exam$^{n.}$ of pp. in Group II.—Removed from the filter into a porcelain dish and boiled with KHO, filtered :—

<table>
<tr><td rowspan="4"><i>Filtrate</i> :—
Acidified with HCl, a <i>white</i> milky liquid only:
<i>Abs. of Group</i> II. B.</td><td colspan="4"><i>Pp.</i> removed from <i>filter</i> by a glass-rod into a porcelain dish and boiled with strong HNO_3 as long as any red fumes came off, added dilute B_2SO_4 and stirred well:</td></tr>
<tr><td rowspan="3"><i>No pp.</i> :—
<i>Abs. of Hg and Pb.</i></td><td colspan="3">Added to sol$^{n.}$ excess of AmHO: blue solution (<i>Pres. of Cu</i>) :—</td></tr>
<tr><td rowspan="2"><i>No pp.</i> :—
<i>Abs. of Bi.</i></td><td colspan="2">Acidified the blue sol$^{n.}$ with HCl and saturated with H_2S; filtered off the black pp. rapidly and boiled it with dilute H_2SO_4:</td></tr>
<tr><td><i>Pp.</i>: dissolved in a little boiling dilute HNO_3, added AmHO in excess, then excess of $H\bar{A}$, then K_4FeCy_6, chocolate-red pp. :—
<i>Pres. of Cu.</i></td><td><i>Filtrate</i>: diluted much and passed H_2S, no pp. :—
<i>Abs. of Cd.</i></td></tr>
</table>

Exam$^{n.}$ of pp. in Group III.a.

Dissolved in a little boiling dilute HCl, added pure NaHO in excess, boiled and filtered :—

<table>
<tr><td rowspan="2"><i>Filtrate</i>: added HCl gradually till the liquid became acid (no pp.), then AmHO in excess, no pp. :—
<i>Abs. of Al.</i></td><td colspan="2"><i>Pp.</i>: dried and fused on platinum foil with Na_2CO_3 and KNO_3, boiled the colourless (<i>abs. of Cr</i>) mass when cold with water; decanted from the undissolved residue :—</td></tr>
<tr><td><i>Residue</i> in the dish dissolved by boiling with a little HCl, added several drops of KCyS: a blood-red coloration:
<i>Pres. of Fe.</i></td><td><i>Sol$^{n.}$</i> being colourless proved abs. of Cr.
Acidified with $H\bar{A}$, boiled, and added $Pb\bar{A}_2$, no yellow pp. :—
<i>Abs. of Cr.</i></td></tr>
</table>

y dissolve, the sol$^{n.}$ was decanted, and a little dilute HCl poured melt. Added a little strong HCl, boiled as long as any smell of sol$^{n.}$ and mixed it with the water sol$^{n.}$ which had been proved

<table>
<tr><td colspan="3">gas, filtered :—</td></tr>
<tr><td colspan="3">s pink (<i>prob. pres. of Co</i>).
some strong HNO_3 and boiled to dryness (the colour of the sol$^{n.}$ changed to light-brown d the residue with HCl, it dissolved completely (<i>Abs. of</i> H_2SiO_3). Added a small portion d; no pp.: <i>Absence of</i> $(PO_4)'''$. To the rem$^{r.}$ added AmCl then excess of AmHO, and</td></tr>
<tr><td colspan="3">te was again pink (<i>vresence of Co</i>): added much Am_2S and boiled, filtered :—</td></tr>
<tr><td rowspan="2"><i>ck pp.</i>
m$^{d.}$ by Table III.B.</td><td colspan="2"><i>Filtrate</i> was yellow (<i>absence of Ni</i>): added Am_2CO_3, filtered :—</td></tr>
<tr><td><i>A white pp.</i>
Exam$^{d.}$ by Table IV.</td><td><i>Filtrate.</i>
Exam$^{d.}$ by Table V.</td></tr>
</table>

EXAM$^{N.}$ OF PP. IN GROUP III.B.

Rinsed the pp. off the filter with some cold dilute HCl, stirred well, filtered :—

<table>
<tr><td rowspan="2"><i>Pp.</i> was black and had already been proved to contain no Ni; fused a portion into a clear borax-bead: bead blue in both flames :—
<i>Pres. of Co.</i></td><td colspan="2"><i>Filtrate:</i> boiled until it no longer smelt of H_2S, added a crystal of $KClO_3$, boiled until the smell of Cl ceased, cooled and added pure NaHO in excess :—</td></tr>
<tr><td>No pp.
<i>Abs. of Mn.</i></td><td>Passed H_2S into the sol$^{n.}$ no pp. :—
<i>Abs. of Zn.</i></td></tr>
</table>

EXAM$^{N.}$ OF PP. IN GROUP IV.

1. *Exam$^{n.}$ by Flame-col$^{n.}$*—Dissolved a small quantity of the pp. in a few drops of HCl upon a watch glass, dipped a loop of Pt-wire into the sol$^{n.}$ and held it in the Bunsen flame: a crimson-red colour was imparted to the flame, which appeared intense red through the indigo-prism: *Pres. of Sr.* This coloration was followed by a yellowish-green very persistent col$^{n.}$: *Pres. of Ba.*

2. *Exam$^{n.}$ in the Wet way.* Dissolved the rest of the pp. in as little boiling $H\bar{A}$ as possible; to a small part of the solution, perfectly cold, added $CaSO_4$ sol$^{n.}$; an immediate pp. formed: *Pres. of Ba.*

To the remainder of the $\overline{HA}$ sol^{n}·, proved to be acid to litmus, added K_2CrO_4 until the liquid appeared yellow, warmed and poured through a double filter :—

<table>
<tr><td rowspan="2"><i>Pp.</i>, which contained all the Ba present, was rejected.</td><td colspan="2">To the clear <i>filtrate</i>, which was orange-red in colour, added AmHO until the colour changed to light yellow, then added Am_2CO_3 in excess and filtered :—</td></tr>
<tr><td><i>Filtrate</i> was rejected.</td><td><i>Pp.</i> : dissolved in as little boiling $\overline{HA}$ as possible; added to a small portion of the sol^{n}· $CaSO_4$ sol^{n}· and boiled, a pp. formed, showing <i>pres. of Sr</i>.
To the remainder of the $\overline{HA}$ solution added H_2SO_4, boiled, filtered, and to the filtrate, which gave no pp. after being boiled with more H_2SO_4, added AmHO in excess and $Am_2C_2O_4$, no pp. :—
<i>Abs. of Ca</i>.</td></tr>
</table>

EXAM^{N}· OF FILTRATE FROM GENERAL TABLE FOR GROUP V.

Evaporated the filtrate to dryness in a porcelain dish, scraped out the residue upon platinum foil, and ignited strongly until white fumes ceased to appear: dissolved the residue off the foil by boiling with water to which several drops of HCl had been added, divided the sol^{n}· into two unequal parts :—

To the *larger portion* added several drops of H_2SO_4 and boiled, then AmHO in excess and several drops of $Am_2C_2O_4$; on heating, a slight pp. formed; filtered, and added to the clear filtrate Na_2HPO_4, a white crystalline pp. :— *Pres. of Mg.*	*Into the smaller portion* dipped a loop of platinum wire, and held it in the Bunsen flame, an intense yellow col^{n}·:— *Pres. of Na,* appearing crimson through the indigo-prism :— *Pres. of K.* Confirmed the pres. of K by stirring a fresh portion of the sol^{n}· with $PtCl_4$ on a watch glass, a yellow pp. formed.

Metals found :—Cu, Fe, Co, Ba, Sr, Mg, Na, K, NH_4.

492. EXAM$^{N.}$ FOR ACID-RADICLES.

The following acid-radicles have already been detected: $(CO_3)''$, $(NO_3)'$, $(ClO_3)'$,.

By reference to the Table of Solubilities (455), under the columns corresponding to the above metals, the only salt insoluble in acids is found to be $BaSO_4$; since Ba is present, and the substance is entirely soluble in acids, (SO_4) *is absent.*

The following acid-radicles have also been proved to be absent in the general examination for metals:—

$(CrO_4)''$, by the HCl solution not becoming green on passing H_2S.

$(AsO_3)'''$ and $(AsO_4)'''$, by no pp. forming in Group II.B.

$(SiO_3)''$ and $(SiF_6)''$, by no residue insol. in HCl remaining on evaporating to dryness with HCl for Group III.A.

$(PO_4)'''$, by testing with $AmHMoO_4$ in the HCl sol$^{n.}$ for Group III.A.

The organic acid-radicles $\bar{A}$ and $\bar{\bar{T}}$ are absent, since there was no smell of burning on heating the substance.

The only commonly occurring acid-radicles remaining to be specially tested for are therefore Cl, Br, I, $(C_2O_4)''$, and $(BO_3)'''$; and of these Br and I are probably absent, since no violet fumes of I or brown fumes of Br were evolved with strong H_2SO_4.

Boiled a portion of the substance with pure Na_2CO_3 sol^n and filtered: acidified portions of the filtrate with—

HNO_3.	HCl.
Added $AgNO_3$; a perfectly white pp. easily sol. in $AmHO$. — *Pres. of* Cl.	*Added* $BaCl_2$; no pp.— *Abs. of* SO_4.
Moistened a piece of turmeric-paper with HCl sol^n of the substance and dried at 100°; no brown stain produced — *Abs. of* BO_3'''.	

492. Found { *Metallic-radicles:* Cu, Fe, Co, Ba, Sr, Mg, Na, K, NH_4.
Acid-radicles: CO_3, NO_3, ClO_3, Cl.

SECTION VII.

APPARATUS, REAGENTS, &c, REQUIRED FOR THE FOREGOING COURSE.

LIST OF APPARATUS REQUIRED BY EACH STUDENT.

494. The following list is so arranged that:—

Division I. contains all apparatus which is required only for the experiments given in Section I.

Division II. is apparatus required both for Section I. and for the Sections on Analysis.

Division III. is apparatus used only in Analysis.

Hence a student intending only to perform the experiments in Section I. will require the apparatus in Divisions I. and II. If he is omitting this portion and intends only to work through the Sections on Analysis, he must be furnished with the apparatus in Divisions II. and III. and will not require that in Division I.

Note.—Apparatus contained in large brackets [ ] is useful but not indispensable: the asterisk marks apparatus which may be kept for general use and need not then be supplied to *each* student.

Division I. (See note at the end of list, page 283.)

*1 Wide-mouthed, stoppered, white glass gas jar; about 24 oz. capacity, and 2 inches inside across the neck, whose upper edge should be ground so as to be closed with a ground glass plate.

*1 Small flanged cylinder, 6 inches in height, 1¼ inches internal diameter, ground at the edge.

*1 Deflagrating spoon, brass cap 2½ inches across.

*1 Small two-necked Woulffe's bottle, not less than 4 oz. capacity; or a small wide-necked bottle of not less capacity.
*1 Small retort, best tubulated with stopper, about 4 to 6 oz. capacity.

DIVISION II.

6 Test-tubes, 5 or 6 inches long and from $\frac{1}{2}$ to $\frac{3}{4}$ inch in diameter.
1 Test-tube cleaner.
2 Lengths of hard glass tubing, rather more than $\frac{1}{8}$th inch internal diameter.
*1 Thistle funnel.
*[1 Clock glass, about 4 inches across.]
1 Round ground-glass plate, 3 inches across.
2 Porcelain dishes, one $2\frac{1}{2}$ inches across, one 3 inches across.
2 Watch glasses, about 2 inches across.
1 Small flask, about 4 oz. capacity.
*Red or black india-rubber tubing; 1 piece 18 inches long and $\frac{5}{16}$ inch internal diameter†, 1 piece 6 inches long and $\frac{3}{16}$ inch internal diameter.
*1 Bunsen-burner † with separate rose-top.
1 Small pestle and mortar.
1 Iron tripod-stand.
1 Piece of wire gauze, 5 inches square.
*1 Round file.
*1 Triangular file.
*1 Small set of cork-borers.
*1 Small retort-stand, 12 inches in height, and fitted with 3 brass rings.

DIVISION III.

4 Glass funnels, two 2 inches across at the top, two $2\frac{1}{2}$ inches across at the top.

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† The Bunsen-burner and gas-tubing may be fixtures on each working bench, and will not then be purchased by each student.

[2 Tobacco-pipe cleaners, for cleaning glass tubes.]

2 Boiling tubes, 6 or 7 inches long, from 1¼ to 1½ inch across.

1 Test-tube stand with at least 12 holes.

1 Nest of 3 or 4 small-sized beakers.

1 Length of glass rod.

*1 Small porcelain crucible about 1¼ inch across, and lid.

1 Strip of platinum foil, 2 inches by 1 inch.

2 Pieces of platinum wire†, each 1½ to 2 inches long.

1 Black's blowpipe.

1 Pipeclay triangle, 2 inches in the side.

*1 Pair of brass crucible tongs.

1 Large flask for wash-bottle, 18 oz. capacity and from 1 to 1¼ inch across the neck.

*1 Wooden filtering-stand.

*1 Tin filter-dryer.

1 Small wicker-basket for draining test-tubes and other glass and porcelain apparatus after they have been washed. Little toilet-baskets, 12 inches in length by 5½ and 4½ inches high, serve well.

Note.—For the experiments with gases the student will also require:—

*A pneumatic trough, or earthenware pan as a substitute for it (see Note, p. 7).

A small piece of candle.

A wax taper, or slips of wood.

A few sound corks of such a size as to fit test-tubes or small flasks.

Caution.—In a laboratory where many students are working, it is best to mark at once each piece of apparatus with initials, number, or with some private mark, so as to be able to recognise it if mislaid. Glass, metal, or porcelain apparatus may be notched with the edge of the three-cornered file, or characters may be scratched upon it with the sharp points made by breaking off the tip of a three-cornered file. Wooden apparatus can be written upon with pen and ink, or scratched with the tip of a penknife.

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† Platinum wire containing Ba is not uncommon: it is known by giving a persistent green colour to the Bunsen flame, and is useless for flame colorations.

APPARATUS FOR GENERAL USE.

495. The following apparatus need not be purchased by each student, but should be kept in constant readiness for general use :—

496. *An Indigo-prism.*—A hollow glass-prism having an extremely small angle at its extremity, with ground-glass stopper fitting water-tight into the neck, is purchased ; this is to be very nearly filled with solution of indigo, and the stopper then tied in securely with fine copper wire. The indigo solution is made either by diluting a solution of indigo in strong sulphuric acid until it has the required intensity of colour*, or by dissolving indigo-carmine in water; the liquid is then allowed to stand for several days to give time for any sediment which may form to settle completely, and when quite clear is decanted into the prism. In the thicker part of the prism the blue must be so intense as to arrest the passage of the light from a flame coloured yellow with a sodium compound, and to cause the potassium flame coloration to appear crimson-red.

497. *Several small slips of cobalt-glass.*—This glass is used for the same purpose as the indigo-prism, but the prism is to be preferred, more especially for the examination for Sr.

498. *Set of cork-borers.*—These are used for perforating a cork into which a piece of glass tubing has to be inserted (8). Nests of borers containing the three or four smallest sizes will be sufficient.

499. *Sulphuretted-hydrogen apparatus.*—It is advisable to employ a large apparatus which will supply this gas to all who are working in the laboratory. The use of a small private apparatus by each student, not only causes unnecessary waste of materials and time, but vitiates the air of the laboratory whilst it is being washed out.

Many forms of apparatus have been devised to supply the gas in a constant stream; probably the following, the simplicity, efficiency, and cheapness of which render it very satisfactory, will be found the most generally useful.

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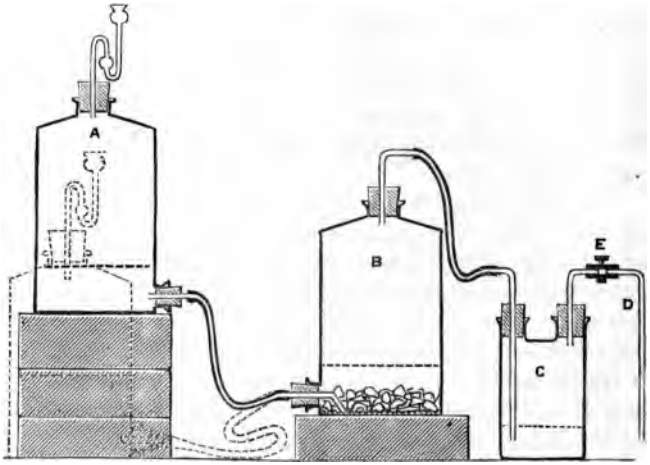
* The addition of the indigo solution to 10 times its measure of acid will usually give the required shade.

Fig. 39.



Note.—The level of the liquids in the vessels is shown by dotted lines; the vessel A when lowered from its stand is also shown in dotted outline. The whole is drawn in section.

500. *Sulphuretted-hydrogen apparatus.*—Two large bottles, A and B, with wide necks, and tubulures near the bottom, and of at least two quarts capacity, are fitted as shown in section in figure 39*. India-rubber stoppers should be used instead of corks, as the latter soon become sodden with acid and rotten, and also lose their elasticity and permit leakage. The vessel B is laid upon its side to prevent breaking the bottom, and lumps of ferrous sulphide are dropped into it: the cork is then inserted, the clamp at E being closed. The glass tube fixed in the lower tubulure of B should be bent downwards, so that its end nearly touches the bottom of the vessel. This drains the acid out of B to the lowest possible level when A is lowered. Strong commercial hydrochloric acid* is poured into A, then an equal bulk of water is added, and the liquids are mixed by shaking the vessel.

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* This apparatus may be purchased completely fitted from Mess$^{rs.}$ Mottershead of Manchester and from most apparatus sellers.

When the gas is required, the vessel A is raised by being placed on a stand of suitable height, the acid runs into B, generates the gas by acting on the ferrous sulphide, and by the pressure of the liquid in A the gas is forced through a little water in the washing-bottle C, and thence through a bent tube at D into the liquid which is to be saturated with sulphuretted hydrogen.

When the flow of the gas is to be stopped, the screw-clamp on the india-rubber joint at E is tightened: by proper management of this screw-clamp the stream of gas can be either totally arrested or controlled to any required extent: this clamp must always be so regulated as to permit of as little waste of gas as possible whilst using the apparatus.

During working hours the vessel A should be kept raised, as shown in the figure; when laboratory work ceases it should be lowered to the position shown in dotted outline. The vessel B is always kept slightly raised by being placed on a wooden block or a brick, so as to encourage the flow of the last portions of acid out of it into A when A is lowered. When the acid is spent, it is easily poured out of A and replaced by fresh: lumps of ferrous sulphide are added as required through the neck of B. The removal of the spent acid and the cleansing of the apparatus should be performed in the open air, or at a sink in a cupboard provided with a good draught.

The neck of A may be kept stopped with a cork carrying a bent funnel whose bend is filled with oil or glycerine; this lessens the escape of gas dissolved in the acid, and tends to prevent the apparatus from causing a smell. If several students require to use the gas at the same time, the broad stopper of B is perforated and fitted with additional tubes and wash-bottles, or a system of distributing pipes or T-pieces connected with the tube from the cork in B may be employed.

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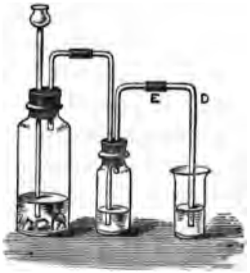
* The use of H_2SO_4 instead of HCl is objectionable, since $FeSO_4$ crystallises in the apparatus, impeding or preventing the action of the acid and its flow through the connecting tube: also it does not produce such an easy and rapid evolution of the gas.

An apparatus for private use may be fitted as shown in fig. 40. The larger bottle contains pieces of ferrous sulphide upon which HCl, diluted with an equal measure of water, is poured through the thistle-funnel: the gas bubbles through a little water in the smaller bottle, and thence into the solution to be saturated.

FIG. 40.



The sulphuretted hydrogen apparatus should stand in a cupboard with a glass front and small doors, which is not used for any other purpose: the cupboard must be furnished with a flue in which a strong draught is created by an argand or ring gas-burner, made of fire-clay or steatite and not of metal; if the cupboard doors are kept closed as much as possible, and students when using the gas are careful to prevent its unnecessary escape, the atmosphere of the laboratory may be kept tolerably free from this badly smelling gas. The cupboard should have within it a drain into which are emptied the spent acid and washings of the apparatus.

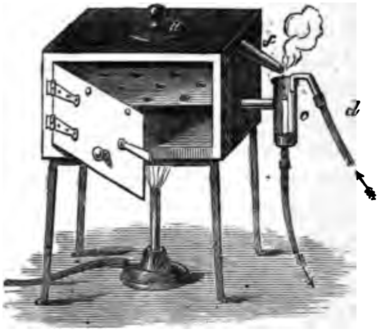
500 *a. Passing sulphuretted hydrogen into a liquid.*— Each student should keep a bent glass tube (D, figs. 39 and 40), made according to the directions in par. 9: when the gas has to be passed into a liquid, the shorter end of this tube is fitted into the india-rubber joint E, and the other end is passed nearly to *the bottom of the liquid;* on slightly opening the clamp E, a stream of the gas will bubble up through the liquid and may be regulated by the clamp; when the gas has passed for about five minutes the liquid will generally be saturated; if this is the case it will smell of the gas strongly after the air above the liquid has been blown out of the vessel and the vessel has been well shaken. Great care must be taken to close the clamp at E completely when the gas is stopped. The tube D must be thoroughly cleansed after use, employing a tobacco-pipe cleaner if necessary for its inside.

501. *A small agate pestle and mortar* (fig. 32, p. 57).—This is required for powdering very hard substances, more particularly minerals. The substance must have been already broken into small fragments, and these are then crushed to powder by pressure and "trituration*," until a powder is produced in which no particles or grains are felt when it is pressed or rubbed with the pestle or the finger; such a powder is commonly termed an "impalpable powder." On no account must a substance be broken by placing it in the mortar and striking it blows with the pestle, as this is very liable to cause the fracture of the mortar.

502. *Several small leaden cups or a platinum crucible* should be kept in readiness for testing for F by paragraph (295) or (296). The leaden cups are easily made by beating out thick sheet-lead into the required shape, the mouth being of such a size as to be readily covered by a watch glass; the small leaden ink pots often employed in school desks serve well for this purpose.

503. *A steam-oven.*—This is a small oven of sheet-copper, heated to the temperature of boiling water (100° C.) by water contained in the space between the exterior of the oven and a copper casing which surrounds it (fig. 41): the outer casing

FIG. 41.



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* Rubbing round and round under the pestle.

often has a circular opening cut in the top, covered by a lid when not in use, which serves as a water-bath (504). The level of water in the jacket is maintained constant by a little contrivance shown in section at *c:* a small reservoir communicating by a lateral tube with the space between the oven and its jacket is constantly replenished by water which drops into it from the supply tube (*d*), the overflow of this reservoir is a tube rising in its centre whose end terminates inside above the level of the lateral tube. The outlet for steam (*f*) may be turned downwards, so that any water formed by the condensation of the steam drops into the reservoir (*c*), or it may be made to communicate with the worm of the still (506), the escape steam being thus condensed into distilled water. The steam-oven should always be supplied with distilled water, as this prevents the formation of a troublesome incrustation in the interior. A vessel of distilled water may be connected with the oven by a syphon, having a bulb or vertical closed tube at its bend, to prevent the air boiled out from the water from stopping the action of the syphon: or the escape pipe (*f*) may be turned upwards and connected with a long vertical or oblique tube which, if the flame is small, will condense the escaping steam, and obviate the necessity of a constant supply of water.

The steam-oven is employed to drive off the moisture from solid substances which require to be dried at a gentle heat.

504. *Several copper water-baths.*—The water-bath consists of a hemispherical copper dish with its edges turned over inwards; a series of flat copper rings of gradually diminishing diameters are made to rest upon the edges of the bath or upon one another's edges, so that the opening at the top can be made as small as may be desired. The bath is about two-thirds filled with water, and is heated on a tripod-stand until the water boils, the excess of steam escaping by a small hole made just below the edge of the bath; the flame should be so regulated that the water is kept gently boiling. A small-sized saucepan is a homely substitute for the copper water-bath.

Any liquid requiring to be evaporated at a gentle heat is placed in an evaporating basin upon this bath, its temperature during evaporation cannot then exceed the temperature of boiling water (100° C.).

The top of the water-oven is frequently made to serve as a water-bath, see (503). If the water-bath requires to be used for any considerable length of time, it may be supplied with water in the same way as the steam-oven (503).

504 *a*. The following cheap and simple device serves the purpose of both water-bath and steam-oven on a small scale. Two circular plates of sheet zinc are obtained*, about four inches in diameter, so as to cover the larger-sized porcelain dishes used by each student: in one of these a circular hole concentric with the edge of the plate and two and three-quarters inches in diameter is cut, in the other a similar hole is made one and a-half inches across. These covers, when placed on one of the larger porcelain dishes, nearly filled with water and boiled by a lamp, convert it into a water-bath, a small dish containing the liquid to be evaporated being supported in the hole of the plates: small quantities of liquid may be evaporated or solid substances may be dried by placing them on a watch glass supported on the smaller perforated plate.

In a large laboratory, however, steam-ovens and water-baths are usually kept constantly heated by steam or boiling water, and are used in common by all students. The steam from a large water-bath may advantageously be condensed to distilled water by connecting the escape-pipe of the bath with a long tube or the still-worm.

505. *Several small tubulated flasks.*—The small flask shown in fig. 38 (p. 132) is very convenient for the reactions described in pars. 260, 264, and 273. It is the form of apparatus best suited for the test for a chloride described in (260), because the $K_2Cr_2O_7$ powder is not introduced through the same tube as the CrO_2Cl_2 escapes by, and the risk of the mixture in the flask spirting into the delivery-tube is rendered very small. Its neck should be closed by an india-rubber or glass stopper.

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* Any working tinman will make them to order.

DISTILLATION OF WATER.

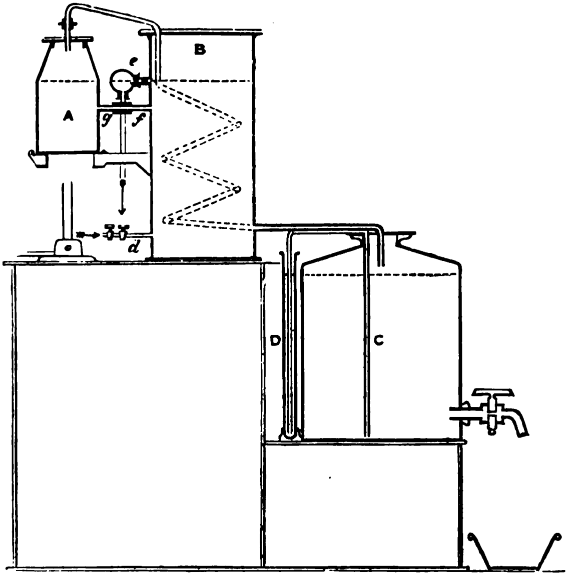
As has been already shown (Exp. 31, p. 24), water ordinarily contains certain solid substances dissolved in it which render it in a chemical sense impure; such water is therefore unfit to be employed for the processes of solution and washing, since any impurity thus introduced into a substance during analysis would be considered when detected to have been present in the original substance. The quantity of these impurities present in any particular water-supply will depend upon the nature of the soil with which the water has been in contact previous to its collection. The water supplied in some districts will be found when subjected to the tests given in [(526) Remark 41] to be almost perfectly pure; carefully collected rain-water will invariably yield no indication of dissolved impurity; the water supplied to the majority of laboratories will, however, be found to be unfit for analytical purposes, until it has been freed from the solid substances dissolved in it by being subjected to the process of distillation.

In most towns distilled water can be purchased, but it is preferable to distil in the laboratory or its immediate neighbourhood all the water required for use. For this purpose the steam obtained either from a steam-boiler or from a copper-still heated by a furnace or gas-burner, or from the water-baths and steam-ovens in the laboratory, is condensed by a tin worm-pipe immersed in a tub through which a constant stream of cold water runs.

506. The following constant apparatus has proved most serviceable in the author's laboratory, and may be used wherever gas and water are laid on in a suitable way. Its advantage lies in the fact that the still is constantly supplied with the hottest portion of the water from the condenser through a lateral tube. When the apparatus has been properly fitted up and the supply adjusted, it requires no attention whatever except lighting and extinguishing the gas under the still, and turning the water supply on and off when distillation is started and stopped: it is advisable occasionally to remove from the interior of the still, and from the supply-tube connecting it with the condensing vessel, the deposit which accumulates by evaporation of the water.

The accompanying sketch (fig. 42) renders a lengthened description of the apparatus unnecessary: it represents the apparatus in section. A copper-still (A) is heated by the flame of a large-sized Bunsen-burner or by a ring gas-burner, the steam passing off from the top of the still through a tin tube bent into a spiral form in the condensing tub (B) which is made of copper or galvanised iron; from this condenser the distilled water flows into a large stoneware vessel (C), with a tap below from which the water is drawn when required. A long syphon-tube (CD) serves as a gauge. The condensing-tub (B) has three tubes let into the side facing the still; one of these (*d*), placed about an inch above the bottom of the vessel, is the inlet for cold water from a cistern whose water

FIG. 42.



level must be higher than that maintained in A and B; this level is marked by the single dotted line, and is kept constant by the overflow-tube *e*, which is connected with a pipe leading to a sink or drain. Just below this overflow-tube is a short tube (*f*), which is on a level with another (*g*), opening into the side of the still; by connecting these two tubes with an india-rubber joint, a supply of warm water from the upper part of the condenser is furnished to the still, keeping the level of water in it constant: a small glass jet drawn out from a piece of glass tubing is fitted into this connecting-tube, and has been found sufficient to prevent a too free circulation of water between the still and condenser. The still is supported on an iron ring, projecting as a bracket from the condenser. By having two taps in the inlet-tube (*d*), or by using two clamps on the india-rubber joint which connects it with the supply-pipe from the cistern, much time is economised, as the flow of water may be regulated once for all by one tap or clamp, the other being used only for stopping and starting the stream. The copper-still should be furnished with a lid screwing upon its mouth for convenience in removing the deposit which collects within, and into this lid should be fastened a brass tube carrying a screw-union by which the tin condensing-pipe may be fastened steam-tight to the still. The wooden stand for the still and storing jar is conveniently fitted with shelves and doors to serve as a cupboard. Such a copper-still, 23 inches in circumference below and 8 inches in height, when filled to a height of 4 inches and heated by the largest-sized Bunsen-burner, yields 2 litres of distilled water per hour.

In laboratories where a larger supply of distilled water is required, a copper still of several gallons capacity may be set in masonry and heated by a small furnace fed with coal or gas. The overflow from the still-tub should then pass into a small cistern whose overflow-pipe keeps its water surface level with that required in the still: and the still and cistern are connected by a tube bent downwards in its middle to prevent the passage of water from the still to the cistern.

For tests of purity see Remark 41 (526).

RECOVERY OF THE METALS FROM SILVER AND PLATINUM RESIDUES.

When $AgNO_3$ or $PtCl_4$ has been added to a solution the liquid is not to be thrown into the sink.

507. *Silver residues.*—If $AgNO_3$ has been added to a liquid it must be emptied into a vessel*, labelled "Silver residues," containing strong HCl which precipitates AgCl. When the vessel is full, the AgCl is allowed to settle, and the liquid decanted from the AgCl; more HCl is then poured in, and the vessel is again ready for use. When a sufficiently large quantity of AgCl has been formed, let it settle, decant off the liquid, and wash the precipitate well by decantation; pour it upon a filter, and dry upon the filter. One of the two following methods may then be employed to obtain metallic Ag from the AgCl:—

1. Mix the AgCl with twice its weight of a mixture in equal proportions by weight of Na_2CO_3 and K_2CO_3; place this in a clay crucible of such a size that the mixture only half fills it, and heat in a furnace until the mass becomes liquid; maintain it in a fused condition for about five or ten minutes, remove the crucible, and tap its bottom several times gently upon a brick to cause the fused particles of Ag to unite. Allow the crucible to cool, break it up, and wash the button of Ag, which will be found at the bottom, until it is quite free from adhering salts.

This button is then dissolved by heating it in a flask with pure strong HNO_3 previously diluted with about half its measure of water; the solution is evaporated to dryness, the residue dissolved in a little water and once more evaporated to complete dryness. The $AgNO_3$ is then dissolved in the right proportion of water to furnish reagent No. 48 (525).

2. A readier method for decomposing the AgCl consists in placing it in a dish with a little water acidified with dilute

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* Most conveniently a large bottle, with a funnel in its neck.

H_2SO_4, and laying upon the AgCl some strips of Zn free from Pb; after some hours the Ag will be separated as a black spongy mass. This is well washed by decantation with hot dilute H_2SO_4, and then washed also by decantation with boiling distilled water till free from the H_2SO_4; the Ag is dissolved in HNO_3 as directed in the first process, the solution evaporated to dryness, and the $AgNO_3$ used for preparing the reagent. Any residue insoluble in HNO_3 will be unreduced AgCl; this may be filtered off and added to the silver residues for subsequent reduction.

508. *Platinum residues.*—Liquids to which $PtCl_4$ has been added are poured into a vessel* labelled "Platinum residues"; when a sufficient quantity has been obtained the liquid and precipitate are poured into a porcelain dish, evaporated to dryness, and heated strongly for some time; as soon as the dish is cool the residue is boiled with water, which is decanted, and upon the residue some solution of oxalic acid is poured; this is evaporated to dryness and the residue strongly ignited. The residue of metallic Pt is once more washed with boiling water, and is then dissolved by heating it with HCl to which one-third its measure of HNO_3 has been added; the solution is evaporated to dryness, the last part of the process being performed over a water-bath. HCl is then poured in, and it is once more evaporated to dryness and heated for some time on the water-bath; this residue when dissolved in water forms the reagent No. 28 (525). Any waste scraps of platinum foil and wire should be carefully preserved, and after having been cleansed by boiling them with HNO_3 and washing well with water, they may be dissolved in HCl and HNO_3 and solution of $PtCl_4$ prepared as is described above.

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* Conveniently a large glass bottle, with a funnel in its neck.

DIRECTIONS FOR PREPARATION OF REAGENTS.

Note.—The reagents required for chemical analysis are much more readily obtained at the present time than they were in former years. They can usually be purchased in a pure state, and therefore processes of preparation, which formerly of necessity occupied a large portion of Manuals of Practical Chemistry, are here omitted. It will be found usually more economical to purchase pure chemicals than to prepare them.

509. Many reagents are required in a dissolved or diluted state; it is a matter of much importance that these solutions should be made of a proper strength, and that the methods of preparing them should be as rapid and simple as possible.

The strength of the solutions is chosen somewhat arbitrarily; indeed, the strength of a solution may be varied with advantage according to the different purposes for which the reagent is to be employed. It is found, however, that in practice a solution of one strength may usually be made to serve all analytical purposes. The proportions of liquid or solid to be mixed with or dissolved in a certain quantity of water, which are given hereafter, are those (or very nearly those) recommended in standard works on analysis. It is not intended, however, to assert that they are for *all* purposes the best, but merely that they are generally suitable.

510. A good and economical store bottle for liquid reagents is the "Winchester quart" bottle, a stock of which generally accumulates in a laboratory, being continually introduced filled with strong acids. The following methods of filling these have been found simple and easy of execution.

The process of preparing solutions divides itself naturally into the "dilution of liquids" (511), and the "solution of solids" (512).

511. DILUTION OF LIQUIDS.

In the following lists the proportions by measure are stated in which the liquid and distilled water are to be mixed; these proportions are roughly, but sufficiently accurately, obtained by measuring with a foot-rule the height of the bottle outside from the bottom to the projecting ridge at the shoulder, then dividing this height in the required proportions, and marking the point of division by a line scratched upon the bottle with a three-cornered file. Thus dilute HCl (No. 2, 524) is made by mixing strong HCl with three times its measure of water; supposing, then, the measured height of the "Winchester" to the shoulder to be 8 inches, a file mark is made 2 inches from the bottom, and on filling the bottle to this mark with strong HCl, then up to the shoulder with distilled water, stoppering and shaking up, a dilute acid of the required strength is obtained. This method serves for numbers 2, 3, 4, 6, and 7 (524). For the preparation of No. 1 see column of remarks in the same paragraph.

512. SOLUTION OF SOLIDS.

For dissolving solids two methods are here described; the former (514) is useful when the solution has to be rapidly prepared, the latter (515) requires less attention and may be used when the solution is not required at once.

513. Against each reagent prepared from a solid there is placed, in paragraph (524) a number, signifying the number of grams (see 560 *et seq.*) to be dissolved in 1 Winchester quart of water, and in all the lists will be found the proportion by weight of solid to water. The former number is obtained from the proportion stated in the next column, by considering that an average-sized "Winchester," filled an inch above the ridge round its shoulder, holds about 2400 c.c., and since each c.c. (560 *et seq.*) of water weighs approximately 1 gram, we have at once the weight of water (2400 grams) contained in the bottle, from knowing its capacity; and the weight of water being known, the weight of solid to be dissolved in it is readily calculated from the proportion given *.

Thus the proper strength of AmCl solution (No. 5, 524) is 1 of solid AmCl dissolved in 8 of water, as shown in the fifth column, or a given weight of water must have dissolved in it $\frac{1}{8}$th its weight of AmCl; since a "Winchester" holds 2400 grams of water, we must then dissolve $\frac{2400}{8} = 300$ grams of AmCl in it to obtain a solution of proper strength.

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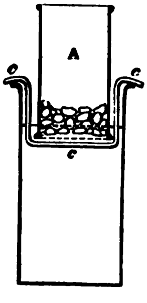
* This method may also be employed when smaller quantities of the reagent are to be prepared. Measure the number of c.c. required to fill the bottle, count the number as grams, and calculate the proportion of solid required as above.

514. Method I.—Weigh out the required quantity of the solid substance, dissolve by heating it with distilled water in a large-sized porcelain dish placed on a tripod-stand over a rose-burner; this solution would often crystallise on cooling, hence it should be at once poured into some cold distilled water contained in a large beaker marked to show the quantity of liquid required to fill the "Winchester." Whilst pouring off the liquid press a wet glass rod, held vertically, against the lip of the dish, and let the bottom of the dish touch the inside of the top of the beaker. These directions will, with proper care, prevent the liquid from running down outside the beaker. Dilute this up to the "Winchester" mark with distilled water, stir well, and if turbid let stand covered from dust until clear; then pour off into the store bottle, carefully leaving the sediment behind. If the liquid is wanted in haste, it may be filtered into the bottle. The coarse brown french filter-paper is useful for this purpose, being strong and porous. It is best to filter the liquid before diluting it to the full extent, as the quantity to be passed through the filter is thus lessened and time economised.

The above method is of general application in the solution of solids.

Fig. 43.



515. Method II.—The following method is advantageous, because the process of solution once started proceeds continuously without further attention. It depends on the fact that the specific gravity of water becomes higher the greater the quantity of solid dissolved in it; hence if the solid is immersed in the water, and supported near the surface, a circulation of the liquid is produced by the constant sinking of the water which has been in contact with the solid and by dissolving some of it has increased in specific gravity, this being replaced by the comparatively lighter liquid from below. The circulation of water over the solid gradually dissolves it without the use of heat, and without requiring any attention after the process has once been started.

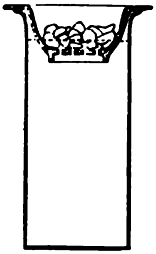
A glass cylinder (A, fig. 43) open at both ends, has one end covered with a piece of muslin, shown by a fine dotted line, which is fastened on by an india-rubber ring, embracing its edges: this cylinder, with a muslin bottom, is supported in a large beaker by means of two pieces of glass rod or tube, bent as shown at *c c c;* the cylinder stands at such a height that the muslin bottom is about two inches below the mark made on the beaker to show the Winchester-quart measure. Distilled water is then poured into the beaker until it buries the muslin to the depth of about half an inch; the weighed substance, best in small pieces, is placed in the cylinder, and the whole allowed to stand until the solid has been dissolved: with large quantities of substance this will usually require several hours, with smaller quantities about twenty minutes or half an hour.

The processes of solution and filtration are performed simultaneously by laying a piece of filter-paper upon the muslin before fastening it on the cylinder; the filter-paper will retard the process, but the coarse grey kind referred to above will do so least.

Perhaps the most advantageous way of using this process, especially for dissolving large quantities of substance, is to start it over night; the solution will then be completed by the morning. In this case the filter-paper will be unnecessary, as any fine particles which have passed through the muslin will have settled, and the liquid can be decanted from them.

It must be borne in mind that the solution of many salts in water, especially such salts as contain water of crystallisation, increases the volume of the water; hence the quantity of water placed in the beaker must be less than the Winchester-quart measure, and the liquid is made up to the required volume, if necessary, after solution has been effected.

FIG. 43 *a*.



As a convenient substitute for the glass cylinder, A, with muslin bottom, an earthenware colander may be employed (fig. 43 *a*), filtration being effected when necessary by laying a piece of filter-paper inside the colander before placing in it the fragments of the substance. Should the beaker be able to contain more than the Winchester-quart of water, a stoppered bottle of suitable size may be immersed in it to raise the water-level sufficiently to reach the substance.

PREPARATION OF SOLUTIONS REQUIRED FOR TRYING THE REACTION OF METALS AND ACID-RADICLES, SECTION IV.

516. The solutions required for trying these reactions should be prepared of a strength suited to yield them in a characteristic manner; it is therefore advisable that solutions of the requisite strength should be kept in readiness, since, if the student has to dissolve some of the solid substance himself, he is liable not to obtain satisfactory results, either because his solution is made inconveniently strong, or because it is too dilute. The plan of keeping these solutions in stock is also recommended by the consideration that it economises the student's time, and tends to prevent waste of chemicals.

517. In paragraphs (529) and (530) are lists of the solutions which are required for the complete series of reactions in Section IV.: the first column gives the number, the second the name, and the third the formula of each substance; in the fourth is stated the number of grams which are to be dissolved in one Winchester-quart of water or acid; in the fifth column the proportion by weight of solid to water, from which the number in column 4 was derived; and in the sixth column will be found special remarks relating to the preparation of particular solutions. The general methods of preparing solutions have already been given in paragraphs (514) and (515).

Pure solid chemicals are almost invariably sold in the crystalline condition, since they have been as a rule purified by the process of crystallisation. The weights given below are therefore weights of the crystalline substance; in a few cases the substance was weighed in the uncrystalline condition—these are marked by an (*a*) placed after the name in the first column, signifying "amorphous." Directions for labelling the store-bottles of these solutions will be found in paragraphs (522) and (523).

LIST OF THE REAGENTS.

518. Paragraphs (524), (525), and (526), contain lists of the reagents, solid and in solution, which should be kept in readiness in the laboratory; they are dissolved in distilled water unless it is otherwise stated.

The reagents are here divided into two classes. The first (524) being in constant use and comparatively inexpensive, should be contained in small bottles placed above each working bench, or one complete set should be provided for the use of each student. The second class (525, 526) are less frequently required, or are of a more costly nature, and one set may be placed in an accessible part of the laboratory for the use of many students.

519. Each reagent is numbered in the following lists; this number is a convenient means of reference and also serves to keep the reagent bottles always standing in the same order, which much facilitates finding any bottle when wanted; the student is recommended to keep his bottles arranged in the order indicated by these numbers. The full name and chemical formula accompany each number, and every label in the laboratory should carry legibly the number, name, and formula of the reagent it contains.

520. In the last column of each list are placed remarks as to the preparation and the condition of the reagent. The numbers found in the last column but one express the strength of the solution of the reagent if a liquid, by stating the proportion of the strong liquid or solid as purchased to the water with which it is mixed or in which it is dissolved, the proportion being expressed as a ratio in which the water always stands last; in the case of liquids the numbers express proportion by measure (m.), in the case of solids the proportion by weight (w.). A number is also placed in the fourth column opposite each reagent prepared by dissolving a solid substance, which denotes the weight in grammes of the solid to be dissolved in a Winchester-quart bottle of water.

Thus No. 2 (524), hydrochloric acid, is prepared by mixing strong HCl with 3 times its measure of distilled water. No. 5, ammonium chloride, is made by dissolving solid AmCl in 8 times its weight of water, or by dissolving 300 grammes of solid AmCl in one Winchester-quart of water. Full descriptions are given in paragraphs (511), (514), and (515), of the methods used for making these solutions.

521. The special reagents which stand on shelves above the bench are kept in small bottles; the solids Nos. 18–21, (524) in 4 oz. wide-mouthed stoppered bottles which are filled when necessary from the store jars (stoneware jars with ground clasp-covers)*, the liquids (Nos. 1–8) in narrow necked 6 oz. bottles and (Nos. 9–17) in 4 oz. bottles supplied from the Winchester-quart bottles in which the store solutions are kept. The general reagents (525, 526) should be kept in bottles of not less than 12 oz. capacity.

522. *Labelling.*—Bottles intended to contain strong acids or alkalis should have labels etched upon the glass, such bottles are easily obtained; an ordinary paper label, however, protected by paraffin varnish, which is applied according to the directions given below (523), will last for a considerable length of time.

The paper labels required for reagents and store bottles are most conveniently kept ready gummed in the form of sheets either separate or stitched together into the form of a book. They must be stored in a dry place. They should bear in bold legible type at least the full name and formula of the substance, the number, if not already printed, being written upon each*. Great care should be bestowed on making the moistened label adhere over its whole surface by gentle pressure with a clean cloth.

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* Powdered solids are conveniently removed from large store jars by means of wooden spoons.

Names or formulæ painted upon the clean glass with "Brunswick black" paint are very permanent, resisting ordinary wear and the action of chemicals perfectly.

523. *Varnishing labels.*—It is advisable to varnish each paper label, which is intended to be permanent, as soon as it is perfectly dry; the label is thus protected against being soiled or wetted. The ordinary process consists in first sizing the label by brushing over it a solution of isinglass in water, and then, as soon as this has perfectly dried, applying a thin coat of label varnish; the bottle must not be used until this varnish is quite hard.

A much more simple process of varnishing consists in melting some solid paraffin, which must not be heated so strongly as to cause it to emit vapour, and brushing this rapidly over the dry label with an ordinary varnishing brush; after a little practice a uniform film can be applied which does not soak into the paper, hardens immediately, and resists the action of water, acids, and alkalis. The chief precaution to be observed is to heat the paraffin as little as possible above its melting-point, as, if it is too hot, it greases the paper.

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* A book of labels ("A Series of Chemical Labels for use in Laboratories") is sold by Messrs Mottershead, of Manchester, which contains the name of each substance in full, printed in clear type, space being left to insert formula and number.

Messrs Mottershead also supply gummed sheets of labels, printed especially for use with this book, containing all the numbers, names, and formulæ in the lists in pars. 524, 525, 526, 529, 530, 531, and 532.

524.—Reagents requ

1. Reference number.	2. Name. [The numbers in brackets refer to remarks correspondingly numbered in the sixth column.]	3. Formula.
Liquids.		
1.	Sulphuric acid (1),	H_2SO_4
2.	Hydrochloric acid (2),	HCl
3.	Nitric acid (3),	HNO_3
4.	Acetic acid (4),	$H\bar{A}$, or $HC_2H_3O_2$
5.	Ammonium chloride (5),	NH_4Cl
6.	Ammonia, or Ammonium hydrate (6)	NH_4HO
7.	Ammonium sulphide (7),	$(NH_4)_2S$
8.	Ammonium carbonate (8),	$(NH_4)_2CO_3$
9.	Ammonium oxalate,	$(NH_4)_2C_2O_4.2H_2O$
10.	Potash, or Potassium hydrate,	KHO
11.	Potassium chromate,	K_2CrO_4
12.	Potassium ferrocyanide,	$K_4FeCy_6.3H_2O$
13.	* Potassium ferricyanide (9),	$K_6Fe_2Cy_{12}$
14.	† Sodium phosphate (10), or Hydric disodic phosphate, or Hydrogen disodium phosphate,	$Na_2HPO_4.12H_2O$
15.	Sodium carbonate,	$Na_2CO_3.10H_2O$ (crys
16.	Calcium sulphate,	$CaSO_4$
17.	Barium chloride,	$BaCl_2.2H_2O$
Solids.		
18.	Sodium carbonate (11),	Na_2CO_3
19.	Borax (12),	$Na_2B_4O_7$
20.	Potassium chlorate,	$KClO_3$
21.	Test papers,	. . .

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* This reagent undergoes decomposition by exposure to light and must not b

† Hydrogen sodium ammonium phosphate $(NaAmHPO)_4$ solution serves bet given the above for Na_2HPO_4.

EACH BENCH.

4. ight of solid in rams to be dis- olved in one “ Winchester ”. of water.	5. Proportion of solid by weight (w.) and liquid by measure (m.) to water.
. .	1 : 5 m.
... ...	1 : 3 „
. .	1 : 3 „
... ...	2 : 1 „
300 . .	1 : 8 w.
... ...	1 : 3 m.
. . .	1 : 1 „
400	1 : 5 w.
100 . .	1 : 24 „
300	1 : 8 „
200 . .	1 : 12 „
200	1 : 12 „
200 . .	1 : 12 „
200	1 : 12 „
600 . .	1 : 4 „
urated solution (527).	
200 . .	1 12 „
small crystals.	
ue and red litmus and turmeric papers in small strips.	

6.

Remarks.

[The small numbers refer to corresponding numbers in the second column.]

1. Pure strong H_2SO_4 must be poured in the proper proportion into water contained in a large thin beaker or an earthen jar, constantly stirring the water all the time: the right proportions are obtained by measurement of the height of the vessel (511). The hot liquid is cooled by immersing the vessel in cold water, and is then poured into the store bottle. If the acid is pure, no white precipitate ($PbSO_4$) forms on dilution.
2. Must be colourless, and give after dilution no precipitate with either $BaCl_2$ or H_2S.
3. Must give no precipitate after dilution, with $BaCl_2$ or $AgNO_3$ added to separate portions.
4. Must give no precipitate with $BaCl_2$.
5. Must give no precipitate or coloration with Am_2S.
6. Must give only a very slight precipitate with lime water, no coloration with Am_2S, and separate portions acidified with HNO_3 must give no precipitates with $BaCl_2$ and $AgNO_3$. “Liquor ammoniæ fortissima,” of 880° specific gravity, is bought.
7. Must be yellow, and give with acids H_2S and a white precipitate of S: it must give no precipitate with solutions of Ca- or Mg-salts.
8. The solid Am_2CO_3 is dissolved by being heated with water in a porcelain dish (514): in diluting, one-fourth of the “Winchester” must be filled with strong AmHO.
9. Must give no blue precipitate with Fe_2Cl_6 solution.
10. Must yield no precipitate with AmHO.
11. Dry finely-powdered solid. It must, after having been dissolved in excess of HNO_3, give no precipitate with $BaCl_2$ or $AgNO_3$, or $AmHMoO_4$ (277), and if evaporated with excess of HCl must leave no residue insoluble in dilute HCl (290 *a*).
12. The borax is best dried by heating in a platinum or porcelain dish until after melting it has again become solid; this solid mass is then finely powdered in a mortar and kept in a stoppered bottle.

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a window.

ipitation of Mg than does Na_2HPO_4 solution: it is prepared of the strength

525.—Reagents for Genera

Note.—Except in large laboratories it will be unnecessary to k in the laboratory, may be made up to the right strength as soon a

1. Reference number.	2. Name. [The small numbers in brackets refer to remarks correspondingly numbered in the fifth column.]	3. Form
25.	Sulphuric acid (17)	H_2SO_4
26.	Hydrochloric acid (2)*	HCl
27.	Nitric acid (3)*	HNO_3
28.	† Platinum chloride	$PtCl_4$
29.	Alcohol, Rectified spirit (18)	C_2H_6O
30.	Tartaric acid (19)	$H_2\overline{T}$ or H_2C
31.	Acid sodium tartrate, Hydric sodic tartrate (20)	$NaH\overline{T}.H_2O$ $NaHC_4H_4O_6$
32.	Slaked lime (21)	$Ca(HO)_2$
33.	Acetic acid (1)*	$H\overline{A}$ or HC_2
34.	Cobalt nitrate (22)	$Co(NO_3)_2.6H$
35.	Hydrofluosilicic acid (23)	H_2SiF_6
36.	Sulphuretted hydrogen solution Hydrogen sulphide (24)	H_2S-solutio
37.	Oxalic acid	$H_2\overline{O}.2H_2O$ $H_2C_2O_4.2H_2$
38.	Potassium sulphocyanide	KCyS
39.	Potassium nitrate	KNO_3
40.	Lead acetate (25)	$Pb\overline{A}_2.3H_2O$ $Pb(C_2H_3O_2)_2$
41.	Sodium acetate	$Na\overline{A}.3H_2O$ $NaC_2H_3O_2.3$
42.	Potassium cyanide (26)	KCy or KC
43.	Bromine-water (27) ‡	Br-water
44.	Stannous chloride (28)	$SnCl_2.2H_2O$
45.	Copper (29)	Cu
46.	Zinc (30)	Zn
47.	Steel (31)	Fe
48.	Silver nitrate	$AgNO_3$
49.	Sulphurous acid	H_2SO_3
50.	Copper sulphate	$CuSO_4.5H_2O$
51.	Magnesium sulphate	$MgSO_4.7H_2O$
52.	Mercuric chloride	$HgCl_2$
53.	Gold chloride	$AuCl_3$

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* See last column on the preceding page.

† Must be used in very small quantity only, being an expensive reagen

‡ Sodium hypochlorite (Na_2Cl_2O), the "Liquor Sodæ Chlorinatæ" of may be used, but does not keep well.

OR THE DETECTION OF METALS.

gents in store in quantity. The 12 oz. bottles in which they are contained pty by the proportions stated below in column 4.

4. oportion by weight of olid to water: and eight in grams for a 12 oz. bottle in square brackets.	5. Remarks. [The numbers in this column refer to those in brackets in the second column.
Strong pure. „ „ „ „ 1 : 30 [15 grams]. Strong. 1 : 10 [45 grams]. Solid. Strong. 1 : 12 [40 grams]. See (528. 4). See (528. 1). 1 : 12 [40 grams]. 1 : 100 [5 grams]. Solid. 1 : 12 [40 grams]. Solid. Solid. 1 : 12 [40 grams]. 1 : 20 [30 grams]. See (528. 2). 1 : 12 [40 grams]. 1 : 12 [40 grams]. 1 : 20 [30 grams]. 1 : 30 [15 grams].	17. Must be colourless and form no brown ring when poured below some $FeSO_4$ solution (247). 18. Strong rectified methylated spirit, which must leave no residue on evaporation, and remain clear on dilution. 19. The solution should be mixed immediately after preparation with several drops of carbolic acid to prevent a vegetable growth forming in it: or better still some crystallised $H_2\bar{T}$ should be dissolved when wanted. 20. Dissolve 10 grams of $H_2\bar{T}$ in 100 c.c.* of water, divide this solution into equal parts, exactly neutralise (35) one part by heating it nearly to boiling and stirring in solid Na_2CO_3 in powder; add to this the other part, cool and dilute to 150 c.c. A few drops of carbolic acid should be added to the solution. 21. Pieces of freshly-burnt lime are placed on a plate, and water is poured upon them until they begin to appear moist on their surface; the superfluous water is then drained off, and as soon as the lime has crumbled to powder, the powder is placed in a broad-mouthed stoppered bottle. 22. The bottle should be closed with a loosely-fitting india-rubber stopper, perforated, and with a glass tube passed through it and dipping into the solution; when a drop of the solution is required, the upper end of the glass tube is closed by the finger, and a drop is delivered from the opposite end by slightly relaxing the pressure of the finger. 23. The solution must give no precipitate with $Sr(NO_3)_2$ solution. 24. This solution must not darken on adding AmHO. 25. A little $H\bar{A}$ must be added to this solution to make it clear. 26. Solution of KCy is made (1 : 12); it decomposes so readily that the solution is best made immediately before use by heating a small piece of solid KCy with distilled water. 27. A few drops of Br dissolved by well shaking with water. 28. Crystals of $SnCl_2$ should be dissolved by heating them with water containing some HCl; the solution should be kept in a well-stoppered bottle containing pieces of granulated or block tin. 29. In strips cut from thin copper-sheet. 30. In strips or rods, or granulated; it must be proved to be free from As by (181, 182, or 183). 31. Common knitting-needles broken into short lengths, and kept in a bottle containing pieces of quicklime to prevent rusting.

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* c.c. is the contraction for cubic centimetre. See weights and measures (560).

526.—REAGENTS FOR GENERAL

Refer to note

1. Reference number.	2. Name. [The small numbers in brackets refer to corresponding numbers in the fifth column.]	3. Formula.
57.	Lime water	$Ca(HO)_2$
58.	Lead acetate in potash (32)	$Pb\bar{A}_2+KHO$
59.	Potassium dichromate	$K_2Cr_2O_7$
60.	Chlorine water	Cl-solution
61.	Ferric chloride (33)	Fe_2Cl_6
62.	Ferrous sulphate (34)	$FeSO_4.7H_2O$
63.	Potassium iodide	KI
64.	Starch (35)	... ...
65.	Indigo-solution (36)	
66.	Manganese dioxide (37)	MnO_2
67.	Potassium dichromate (38)	$K_2Cr_2O_7$
68.	Ether (methylated)	$(C_2H_5)_2O$
69.	Carbon disulphide	CS_2
70.	Potassium nitrite (39)	KNO_2
71.	Nitrogen tetroxide solution	N_2O_4-solution
72.	Ammonium molybdate (40)	$(NH_4)HMoO_4$
73.	Calcium fluoride	CaF_2
74.	Potassium disulphate	$KHSO_4$
75.	Microcosmic salt, or Hydric ammonic sodic phosphate (40 a)	$NaAmHPO_4.4H_2O$
76.	Wax, or paraffin	... ...
77.	Calcium chloride	$CaCl_2.6H_2O$ (cryst
78.	Potassium chloride	KCl
79.	Marble	$CaCO_3$
80*.	Distilled water (41)	H_2O
81.	Pure sodium hydrate (42), pure soda	NaHO
82.	Fusion mixture (43)	$Na_2CO_3+K_2CO_3$
83.	Barium carbonate (44)	$BaCO_3$
84.	Solution of sodium acetate in dilute acetic acid (45)	$Na\bar{A}+H\bar{A}$
85.	Sodium nitrate (46) (solid)	$NaNO_3$
86.	Silica, or finely powdered white sand. (See 548. 4.)	SiO_2
87.	Hydrofluoric acid (47)	HF
88.	Barium hydrate (in crystals)	$Ba(HO)_2.8H_2O$
89.	Zinc sulphate. (See No. III.)	$ZnSO_4.7H_2O$

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* Nos. 80–89 are certain sp

B THE DETECTION OF ACID-RADICLES.

heading of (525).

4. Proportion by weight of solid to water: and weight in grams for a 12 oz. bottle. in square brackets.	5. Remarks. [The numbers in this column refer to corresponding numbers in the second column].
See (527). 1: 24 [20 grams]. See (528. 3). 1: 24 [20 grams]. Solid. 1: 60 [8 grams]. Solid. Solid. Solid. 1: 24 [20 grams]. See (528. 5). In powder. In small pieces. In small crystals. Solid in pieces. 1: 12 [40 grams]. 1: 12 [40 grams]. In pieces as large as a pea.	32. To some of the $Pb\bar{A}_2$ solution (No. 40) KHO solution is added until, on warming, the precipitate at first formed is just redissolved: paper dipped into this liquid is a very delicate test for H_2S (230). 33. The solution should not contain any free acid: to remove acid AmHO is added until the further addition of *a single drop* gives a reddish-brown precipitate of Fe_2HO_6 in the solution. 34. $FeSO_4$ solution rapidly oxidises in the air, hence the $FeSO_4$ should be kept in the solid state as small green crystals, which should not show a yellow coating in any part; these are dissolved when required by crushing and shaking with cold water. 35. Starch solution rapidly changes, it is best therefore to keep the starch as a powder. Starch solution is made by stirring 2 grams of this powder, which has been made into a paste with 10 c.c. of cold water, into 100 c.c. of boiling water and cooling. 36. Made by dissolving indigo-carmine in water. 37. Should be kept in fine powder; it must not evolve Cl or CO_2 when warmed with strong H_2SO_4. 38. In small pieces, or in powder. 39. The solution is prone to change, and should be made in small quantity only: it must evolve copious red fumes when mixed with H_2SO_4. 40. Make the solution with the following proportions:—1 gram of Am_2MoO_4 is dissolved in 12·5 c.c. of strong AmHO which has been previously mixed with an equal quantity of water, the solution is allowed to stand (if necessary) till clear, then poured off into 25 c.c. of strong HNO_3: the liquid will become hot, and should be allowed to stand until it is cool before being used.

40 *a*. The HNO_3 solution of this salt must yield no precipitate with $AgNO_3$.

41. Must leave no residue on evaporation; in separate portions no precipitates must be caused by $BaCl_2$, $AgNO_3$, or $Am_2C_2O_4$, neither must any precipitate or even dark coloration be produced by addition of Am_2S.

42. The proportion by weight should be 1: 10 of water; it is best kept in green glass bottles, as it slowly dissolves Pb from white flint glass. The solution must not become dark in colour when mixed with H_2S, nor give a gelatinous precipitate (Al_2Ho_6) when mixed with excess of AmCl solution.

43. Dry finely powdered Na_2CO_3 and K_2CO_3 are intimately mixed in the proportion of 53: 69 by weight, and kept in a stoppered bottle.

44. Pure powdered $BaCO_3$ is either purchased, or is made by precipitating $BaCl_2$ solution completely with Am_2CO_3 or Na_2CO_3 solution, and washing the precipitate well by decantation. This powder is then mixed with distilled water to the consistency of thin cream.

45. Dissolve 20 grams of $Na\bar{A}$ in 60 c.c. of distilled water, and add to the solution 40 c.c. of strong $H\bar{A}$.

46. By neutralising strong hot Na_2CO_3 solution with HNO_3 and evaporating to dryness. Sold also as Chili saltpetre.

47. Purchased and kept in gutta-percha bottles.

reagents entered apart.

PREPARATION OF SATURATED SOLUTIONS.

By a "saturated solution" is meant a solution containing the maximum quantity of the substance which the solvent can dissolve: this quantity varies with the temperature of the solution: the solutions spoken of in this book are saturated at the ordinary temperature.

PREPARATION OF SATURATED SOLUTIONS OF SOLIDS.

527. The method described in (515) may be used, or the substance in powder may be constantly shaken up with the solvent for some time, and the solution then separated from excess of the solid by decantation or filtration. The most ready method of preparing a saturated solution of $Ca(HO)_2$, or of $CaSO_4$, is to pour some of the powder into a Winchester-quart, then fill it up to the shoulder with water, and mix the powder thoroughly with the water by shaking the bottle; the shaking is repeated at intervals during half an hour or more; the bottle is then allowed to stand until the excess of powder has settled and the liquid above it is quite clear, when the liquid is poured off into another "Winchester," leaving the powder behind; fresh water is poured upon this powder and more solution made as above by constant shaking, the bottle being allowed to stand by until the fresh supply is required. By thus making a fresh stock of solution whenever one lot is decanted, time is allowed for the freshly prepared solution to become perfectly clear before it is required for use.

1. *Lime-water* is made in the above manner by shaking slaked lime in powder with common *tap-water.*

2. *Calcium sulphate* solution is prepared by saturating *distilled water* by the above means with gypsum or plaster of Paris in powder.

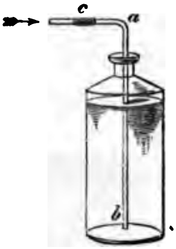
PREPARATION OF SATURATED SOLUTIONS OF GASES.

528. The gas is made to bubble in a constant stream from the end of a glass tube which dips nearly to the bottom of the liquid (fig. 44); the liquid must be kept cold, and be contained in a bottle which is fitted with an accurately ground stopper. In order to ascertain whether the solution is saturated, the bubbles of the gas are occasionally watched to see whether they diminish in size as they rise through the liquid; if they do not, the bottle, after being closed tightly by the thumb or by inserting the stopper, is violently shaken; if this causes a pressure and escape of gas from the bottle into the air on unclosing its mouth, the liquid is saturated; if, on the contrary, it causes a pressure of air into the bottle, the gas must be passed again for some time, and the trial repeated, until on shaking as directed above, an outward pressure is noticed.

Note.—All the preparations mentioned below should be made in a draught cupboard, as the gases are injurious to the lungs if inhaled.

1. *Sulphuretted hydrogen solution* is made by fitting the bent tube *a b* (fig. 44) by means of the india-rubber joint *c*, upon either of the apparatus described in (500) for the preparation of H_2S; the gas is thus made to bubble through the distilled water (best recently boiled to expel air, and cooled) until it is saturated. The solution should not be made in large quantities at a time, as it gradually decomposes, depositing sulphur and losing its smell, it is then unfit for use.

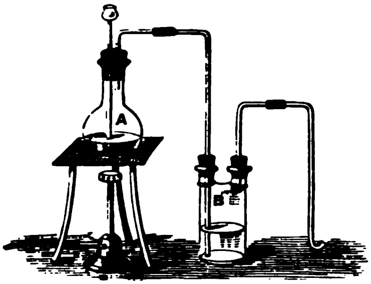
FIG. 44.



2. *Sulphurous acid solution* is prepared by passing sulphur dioxide gas into distilled water until it is saturated. The gas is made by heating scraps of copper with strong H_2SO_4 in a glass flask fitted as is shown in A (fig. 45). The gas passes from the flask through some water contained in a washing bottle as shown at B (fig. 45), and from this bottle into the distilled water (fig. 44) through the tube *a b*, which is connected with the outlet tube of the washing bottle by means

of the india-rubber joint *c*. The solution must be kept in a well-stoppered bottle.

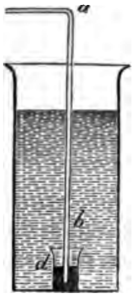
FIG. 45.



3. *Chlorine-water.*—Chlorine gas is made in the apparatus shown in fig. 45, by gently heating manganic oxide in lumps or powder, in the flask A with some strong commercial hydrochloric acid, previously mixed with a third its volume of water. The gas bubbles through a little water in the washing bottle B, and thence through the bent tube *a b* into the distilled water (fig. 44). This solution must be kept in a dark place, or in a bottle which is coated with black paper, as it undergoes alteration by light.

4. *Hydrofluosilicic acid* is made by passing silicon fluoride gas into water. The gas is prepared in a flask fitted as shown in A, fig. 45. Into this flask there is first poured an intimate mixture of 50 grammes of dry, pure, finely powdered fluor-spar with 50 grammes of fine white sand; 300 grammes of strong H_2SO_4 are then poured into the flask through the funnel tube, and the acid is mixed with the powder by shaking it round in the flask; the gas is caused to be evolved by gently heating the flask, and is made to pass first through the bottle B (fig. 45), which must be empty and dry inside; thence it escapes through a bent tube *a b* (fig. 46), which is fitted upon the outlet tube of B by an india-rubber joint: the end of the tube, *a b*, is made to dip

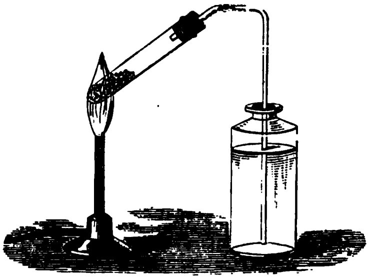
FIG. 46.



into mercury contained in a small beaker *d* (fig. 46), which stands in a large beaker containing 400 c.c. of water. As soon as the silicon fluoride gas comes into the water, after escaping from the mercury, it is decomposed into hydrofluosilicic acid which dissolves in the water, and silica which remains suspended in the water as a gelatinous mass; the silica very soon closes the end of the delivery-tube *a b* unless it is kept immersed in the mercury. As the current of gas slackens, the heat is raised, until white fumes of H_2SO_4 begin to appear in the preparation flask; the process is then arrested and the gelatinous silica is separated from the solution by squeezing the latter through fine muslin and afterwards filtering it if not quite clear. The silica may be dried, heated strongly in a porcelain dish, and put by in a stoppered bottle as reagent No. 86 (526), for which it serves admirably.

5. *Nitrogen-tetroxide solution.* — Lead nitrate in dry pieces is heated in a test-tube fitted with cork and delivery-tube (fig. 47), and the red fumes which escape are passed into dilute H_2SO_4. Care must be taken to maintain the heat uniformly after the fumes begin to be evolved, else the liquid may be sucked back into the hot tube by the contraction of the gas on cooling; also the delivery tube must be removed from the liquid as soon as the heating is stopped.

FIG. 47.



529. SOLUTIONS FOR

Note.—Each bottle should bear on its label the number, name, and formu tions, containing the powdered solid of each of those substances whose nu number, and formula of the substance.

1. Reference number.	2. Name. [The numbers in brackets refer to similarly numbered remarks in the last column.]	3. Formula.
100.s.	Potassium chloride	KCl
101.s.	Ammonium chloride	AmCl
102.s.	Sodium chloride.	NaCl
103.s.	Magnesium sulphate	$MgSO_4.7H_2O$
104.	Barium chloride.	$BaCl_2.2H_2O$
105.	Strontium nitrate	$Sr(NO_3)_2.4H_2O$
106.	Calcium chloride	$CaCl_2.6H_2O$
107.s.	Alum, or Aluminium potassium sulphate	$AlK(SO_4)_2.12H_2O$
108.s.	Ferric chloride (*a*)*	Fe_2Cl_6
109.s.	Ferrous sulphate (48)	$FeSO_4.7H_2O$
110.s.	Chrome alum, or Chromium potassium sulphate	$CrK(SO_4)_2.12H_2O$
111.s.	Zinc sulphate	$ZnSO_4.7H_2O$
112.s.	Manganese chloride (*a*)	$MnCl_2$
113.s.	Nickel sulphate	$NiSO_4.7H_2O$
114.s.	Cobalt nitrate	$Co(NO_3)_2.6H_2O$
115.s.	Mercuric chloride	$HgCl_2$
116.s.	Lead acetate (49)	$Pb\bar{A}_2.3H_2O$
117.s.	Bismuth nitrate (50)	$Bi(NO_3)_3.5H_2O$
118.s.	Copper sulphate (*a*)	$CuSO_4.6H_2O$
119.s.	Cadmium sulphate (*a*)	$CdSO_4.4H_2O$
120.s.	Arsenious oxide (*a*) (51) Solution in dilute HCl	As_2O_3
120.	Arsenious oxide (*a*) (52) Solution in water	
121.s.	Sodium arsenate	$Na_2HAsO_4.12H_2O$
122.s.	Antimonious chloride (53) †	$SbCl_3$
123.s.	Stannous chloride (54)	$SnCl_2.2H_2O$
124.	Stannic chloride (55)	$SnCl_4$
125.	Silver nitrate	$AgNO_3$
126.	Mercurous nitrate (56)	$Hg_2(NO_3)_2.H_2O$

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* *a* signifies that the solid substa

† Solid Sb_2O_3 or $K(SbO)\overline{T}$ (Tartar e

CTIONS OF THE METALS.

ubstance. Small 4 oz. wide-mouthed bottles should be kept near the solu-, small (s) affixed to it; these bottles should also carry on their label the name,

4. eight of solid in s to be dissolved in e “Winchester.”	5. Proportion by weight of solid to water.	6. Remarks. [These numbers correspond to the small reference numbers in brackets in the second column.]
100	1 : 25	48. The solution must be made acid with a little H_2SO_4, and some clean iron nails kept in it, or better, a small quantity is freshly prepared when required (p. 309. 34, col. 5). 49. A little $\overline{HA}$ must be added to this solution to make it clear. 50. 25 grams of $Bi(NO_3)_3$ must be dissolved by heating with 25 c.c. of strong HCl diluted with 25 of water; this solution is cooled and poured into the bottle, and the measure made up by pouring in dilute HCl containing one-twentieth of its volume of strong BCl. 51. Dissolve 10 grams of As_2O_3 by heating it with 50 c.c. of strong HCl mixed with 50 c.c. of water, and dilute to the Winchester-quart. 52. Boil excess of As_2O_3 for several minutes with 200 c.c. of water, filter and dilute to the quart‡. 53. 28 grains of crystallised $SbCl_3$ are dissolved in 25 c.c. of strong HCl mixed with 25 c.c. of water, and diluted to the quart‡ with HCl mixed with four times its measure of water. 54. Boil 25 grams of $SnCl_2$ with 50 c.c. of strong HCl mixed with 50 c.c. of water, as soon as it has dissolved to a clear solution dilute to the quart‡. Scraps of Sn must be kept in the bottle. 55. Heat 25 grams of $SnCl_2$ with 25 c.c. of strong HCl and 100 c.c. of water; whilst constantly stirring, add $KClO_3$ to the hot solution until the liquid turns yellow and Cl is smelt, boil off the Cl and dilute to the quart‡. 56. Dissolve by warming 25 grams of $Hg_2(NO_3)_2$ with 6 c.c. of strong HNO_3 diluted with 114 c.c. of water, then pour water into this solution until it is diluted to a quart‡. Keep Hg in the bottom of the bottle.
200	1 : 12	
50	1 : 50	
50	1 : 50	
25	1 : 100	
100	1 : 25	
200 (in crystals)	1 : 12	
200	1 : 12	
25	1 : 100	
25	1 : 100	
50	1 : 50	
25	1 : 100	
25	1 : 100	
50	1 : 50	
50	1 : 50	
50	1 : 50	
50	1 : 50	
25	1 : 100	
50	1 : 50	
25	1 : 100	
10	1 : 250	
50	1 : 50	
25	1 : 100	
25	1 : 100	
25	1 : 100	
25	1 : 100	

the crystalline condition.

be used for the blowpipe reaction. ‡ Winchester-quart.

530. SOLUTIONS FOR TH

Note.—Refer to note at the head of the preceding Table (529), which i
pared amongst the reagents (524, 525, 526), and the solutions for reactions fo

1. Reference Number.	2. Name. [The small numbers in brackets refer to the corresponding numbers in the sixth column.	3. Formula.
130.	Sodium sulphate	$Na_2SO_4.10H_2O$
131.s.	Sodium carbonate (57)	Na_2CO_3
	Marble (calcium carbonate) (57)	$CaCO_3$
132.s.	Ferrous sulphide (58)	FeS
	Ammonium sulphide (58)	Am_2S
133.s.	Acid sodium sulphite	$NaHSO_3$
134.	Sodium hyposulphite	$Na_2S_2O_3.5H_2O$
135.	Sodium hypochlorite (59)	Na_2Cl_2O
136.s.	Potassium nitrite (*a*)	KNO_2
137.s.	Potassium nitrate	KNO_3
138.s.	Potassium chlorate	$KClO_3$
139.s,	Sodium chloride (60)	$NaCl$
140.s.	Sodium bromide	$NaBr$
141.s.	Potassium iodide	KI
142.	Sodium phosphate	$Na_2HPO_4.12H_2O$
143.	Sodium arsenate (61)	$Na_2HAsO_4.12H_2O$
144.s.	Borax (62)	$Na_2B_4O_7.10H_2O$
145.	Potassium bichromate	$K_2Cr_2O_7$
146.s.	Sodium silicate (63)	Na_2SiO_3
	Silica (white sand)	SiO_2
147.s.	Fluor-spar (calcium fluoride)	CaF_2
148.	Hydrofluosilicic acid (64)	H_2SiF_6
149.s.	Potassium cyanide (65)	KCy
150.	Potassium ferrocyanide	$K_4FeCy_6.3H_2O$
151.	Potassium ferricyanide	$K_6Fe_2Cy_{12}$
152.	Potassium sulphocyanide	$KCyS$
153.s.	Ammonium oxalate (66)	$Am_2C_2O_{42}.2H_2O$
154.s.	Tartaric acid (67)	$H_2\overline{T}$
	Sodium tartrate (68)	$Na_2\overline{T}.2H_2O$
155.s.	Sodium acetate (69)	$Na\overline{A}.3H_2O$

REACTIONS OF THE ACID-RADICLES.

also applicable to this. Many of the solutions in this list are already pre-
metals (529).

4. eight of solid in grams to be dissolved in one Winchester-quart.	5. Proportion by weight of solid to water.
. 25 .	1 : 100
... 100 ...	1 : 25
. 100 .	1 : 25
. 100 .	1 : 25
... 100 ...	1 : 25
. 50 .	1 : 50
... 50 ...	1 : 50
. 50 .	1 : 50
... 12 ...	1 : 200
. 50 .	1 : 50
... 50 ...	1 : 50
... 25 ...	1 : 100
In fine powder.	
. 50 .	1 : 50
... 25 ...	1 : 100
. 25 .	1 : 100
... 25 ...	1 : 100
. 100 .	1 : 25
... ...	1 : 10

6.

Remarks.

[The numbers correspond to the small reference numbers in brackets in the second column.

57. The Na_2CO_3 is required only in the solid form, and can be taken from the reagent bottle on the working bench, No. 18 (524).
The marble is kept in small pieces as large as a pea; it is found in reagent bottle No. 79 (526).
58. The FeS is kept as a solid in small pieces as large as a pea.
The Am_2S can be taken from reagent bottle No. 7, on the working bench.
59. Strong "Liquor Sodæ Chlorinatæ," diluted with an equal measure of water.
60. No. 102, paragraph 529.
61. No. 121, paragraph 529.
62. No. 19, paragraph 524.
63. The solution is made by dissolving 100 grams of the thick syrup, sold as "soluble glass," in water, and diluting to the Winchester-quart. The solid substance to be used is sand finely ground.
64. No. 35, paragraph 525.
65. No. 42, paragraph 525. The solution decomposes by keeping, a little solid is dissolved when required.
66. No. 9, paragraph 524.
67. No. 30, paragraph 525.
68. 123 grams of $H_2\overline{T}$ are dissolved by heat in 500 c.c. of water, the hot solution is exactly neutralised with solid Na_2CO_3, and then diluted to a Winchester-quart.
69. A little of the solid substance (see No. 41, 525) is dissolved, when required, in water.

531. CHEMICALS REQUIRED FOR SECTION I.

In the following list those substances which are used for Section I. only are marked by letters of the alphabet. The bottles containing these substances should be arranged apart in alphabetical order. Against others a number is placed; this indicates that the substance is used for analytical purposes, and will therefore be found, together with any necessary descriptive remarks, in one of the foregoing lists (524, 525, 526, 529, 530).

Note.—All numbers below 22 refer to bottles standing on the shelves above the working bench. If a substance is required for more than one experiment, its entry is not repeated.

Letter or number of reference.	Name	Formula.	Remarks.
A.	Mercuric oxide	HgO.	The orange-coloured *precipitated* oxide is best.
20.	Potassium chlorate	$KClO_3$.	—
B.	Manganese dioxide	MnO_2.	Commercial oxide in powder.
C.	Wood-charcoal	C.	In pieces as large as a hazel-nut.
57.	Lime-water	$Ca(HO)_2$.	—
D.	Sulphur	S.	Pieces of roll sulphur as large as a pea.
21.	Litmus and turmeric papers	—	—
59.	Potassium dichromate	$K_2Cr_2O_7$.	—
E.	Phosphorus	P.	Pieces as large as half a pea, kept in a stoppered bottle under water.
F.	Granulated zinc	Zn.	Not necessarily pure.
G.	Hydrochloric acid	HCl.	Strong, commercial.
H.	Marble	$CaCO_3$.	In pieces not larger than a hazel-nut.
I.	Copper	Cu.	Turnings, clippings, or filings.
J.	Nitric acid	HNO_3.	Strong, commercial.
K.	Ammonium chloride	NH_4Cl.	Solid, commercial.
32.	Slaked lime	$Ca(HO)_2$.	—
L.	Oxalic acid	$H_2C_2O_4.2H_2O$.	Solid, in crystals.
M.	Sulphuric acid	H_2SO_4.	Strong, commercial.
N.	Sodium hydrate	$NaHO$.	Strong solution in water.
O.	"Turkey red"	—	In strips.
P.	Sodium chloride	$NaCl$.	In pieces as large as a hazel-nut broken from a lump of rock-salt.
3.	Nitric acid	HNO_3.	
48.	Silver nitrate	$AgNO_3$.	
6.	Ammonium hydrate	NH_4HO.	
9.	Ammonium oxalate	$(NH_4)_2C_2O_4$.	
17.	Barium chloride	$BaCl_2$.	
7.	Ammonium sulphide	$(NH_4)_2S$.	
39.	Potassium nitrate	KNO_3.	

532. CHEMICALS REQUIRED FOR THE EXPERIMENTS ON ANALYTICAL OPERATIONS (SECTION III.)

All the substances, with those exceptions* only which are named in this list, are required either for analytical purposes (see lists in paragraphs 524, 525, 526, 529, 530), where they may be found by the number placed in the first column; or they are already entered on the list of substances required for experiments on the gases (531), where they may be found by the reference letter in the first column. A letter (s) affixed to the number of reference indicates that the substance is required in the solid condition, see note at heading of (529).

Number or letter of reference.	Name.	Formula.
39.	Potassium nitrate	KNO_3.
118. s.	Copper sulphate	$CuSO_4.6H_2O$.
79.	Marble	$CaCO_3$.
2.	Hydrochloric acid	HCl.
45.	Copper	Cu.
3.	Nitric acid	HNO_3.
17.	Barium chloride	$BaCl_2.2H_2O$.
8.	Ammonium carbonate	Am_2CO_3.
161. *	Filter-paper	In sheet, or cut.
1.	Sulphuric acid	H_2SO_4.
A.	Mercuric oxide	HgO.
K.	Ammonium chloride	NH_4Cl.
100. s.	Potassium chloride	KCl.
18.	Sodium carbonate	Na_2CO_3.
112. s.	Manganese chloride	$MnCl_2$.
19.	Borax	$Na_2B_4O_7$.
62. s.	Ferrous sulphate	$FeSO_4.7H_2O$.
102.	Sodium chloride	$NaCl$.
160. *	Wood-charcoal	C.
116. s.	Lead acetate	$Pb\bar{A}_2.3H_2O$.
—	Red cabbage leaves	—
6.	Ammonium hydrate	NH_4HO.
5.	Ammonium chloride	NH_4Cl.
21.	Litmus-paper Turmeric-paper	— —

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* The exceptions are Nos. 160 and 161, which numbers refer to paragraph 533; also the red cabbage leaves, which are readily obtainable.

533. SUNDRY OTHER REQUISITES NOT INCLUDED IN THE PRECEDING LISTS.

Reference number.	Name and description.
160.	*Wood-charcoal.*—This may be purchased in small pieces commonly used for fuel. If required for blowpipe experiments (33), the charcoal should be tolerably free from cracks and from bark. It may be sawn into convenient shape and size by a fine-toothed saw.
161.	*Filter-paper* may be bought in sheets which are cut into squares of the required size; these are folded and cut as directed in experiment 40 (p. 44). Ready cut circular filters can also be purchased, which only require to be folded.
162.	*Corks.*—These should be obtained as free as possible from holes or cracks, they must be sound and easily softened by pressure. All sizes between 1½ inch and ½ inch in diameter should be kept in stock. For wash-bottles and other permanently fitted apparatus in which the cork is not subjected to much heating, the india-rubber stoppers are much to be preferred to corks, since they are almost imperishable and retain their elasticity very much longer than corks do.

SUBSTANCES TO BE GIVEN FOR ANALYSIS BY THE PRECEDING TABLES AND DIRECTIONS.

534. It must be understood that the substances mentioned below are merely brought forward as examples of what may be given to the student for analysis, the teacher will use his own judgment in adopting and extending the list.

535. Substances for analysis whilst trying through the Reactions for Metals and Acid-radicles.

If the student, after working through the reactions for each of the metallic groups, intends to analyse substances containing one member only, or any two or more members, of that group, it is a good plan to analyse first a few substances containing only one member and to gradually increase the number present in those subsequently given. It is well to keep these substances in the dissolved state* as a rule, as they are intended mainly to afford practice in separation and detection according to the group-tables, and the time spent by the student in preparing the solution is therefore wasted. Occasionally, however, a solid substance may be given in which the metal or acid-radicle present is to be detected by blow-pipe tests or other tests made on the solid substance.

As examples of the substances to be given, in the order best suited to the gradual advance in difficulty, the following, selected for groups V., IV., and III.B, will serve :—

Group V.	*Group IV.*	*Group III.b.*
Solution of $NaCl$	Solution of $BaCl_2.2H_2O$	Solution of $ZnSO_4.7H_2O$
" " NH_4Cl	" " $Sr(NO_3)_2.4H_2O$	" " $MnCl_2$
" " KCl	" " $CaCl_2.6H_2O$	" " $NiSO_4.7H_2O$
" " $NaCl+KCl$	" " $Sr(NO_3)_2.4H_2O+CaCl_2.6H_2O$	" " $Co(NO_3)_2.6H_2O$
" " $NaCl+NH_4Cl+KCl$	" " $BaCl_2.2H_2O+Sr(NO_3)_2.4H_2O+CaCl_2.6H_2O$	" " $MnCl_2+ZnSO_4.7H_2O$
" " $NaCl+NH_4Cl$	" " $BaCl_2.2H_2O+Sr(NO_3)_2.4H_2O$	" " $Co(NO_3)_2.6H_2O+NiSO_4.7H_2O+ZnSO_4.7H_2O$
" " $MgSO_4.7H_2O+NaCl+KCl$		&c., &c., &c.
Solids : $NaCl$, NH_4Cl, KCl, $AmCl+NaCl$, &c.		*Solids* : $MnCl_2$, $NiSO_4.7H_2O$, $Co(NO_3)_2.6H_2O$. $ZnSO_4.7H_2O$, &c.

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* The solutions already made for the reactions (529, 530) will serve very well for this purpose, being given either singly or mixed.

536. Substances are given to be tested for the members of an acid-radicle group in the same order (535)*, the first substances containing one member only, those subsequently given containing two or more members of the group.

537. Substances for Analysis by the directions contained in Section V.

Any one of the solutions named in paragraphs 529 and 530 may be given for analysis by Section V., some of them being neutral, some acid, and others alkaline in reaction. The following more difficult solutions with acid or alkaline reaction may also be added to the list:—

Acid.	*Alkaline.*
$Ca_3(PO_4)_2$, "Bone-ash" dissolved in dilute HCl.	$KSbO_3$ dissolved in water.
BaC_2O_4 dissolved in dilute HCl.	Na_2SiO_3, solution of "soluble glass."
$Mg(BO_2)_2$† „ „ HCl.	Na_2SnO_3, "preparing salt" of the dyer.
$BaCrO_4$ „ „ HCl.	

Any of the solid salts which were dissolved in order to prepare the solutions in paragraphs 529 and 530, may be given for analysis as solid substances by (331 *et seq.*).

As examples of substances possessing metallic appearance (366), the following may be mentioned as suitable for analysis:—

Zn, in pieces or filings.
Pb ,, ,,
FeS_2, Iron Pyrites.
NiAs, Kupfernickel.
Graphite, or Black lead.
Iron filings.

As insoluble substances which require to be examined by (367 *et seq.*) the following may be given for analysis:—

$BaSO_4$.
$PbSO_4$.
AgCl.
SnO_2, Tinstone.
CaF_2, Fluor spar.
$FeCr_2O_4$, Chrome Iron Ore.
S, as pieces of roll sulphur.
C, as powdered wood-charcoal or plumbago.

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[*] See foot-note on page 321.

[†] Made by mixing hot solutions of $Na_2B_4O_7$ and $Mg(NO_3)_2$.

538. Solid Substances to be analysed by the Preliminary Examinations.

For the Preliminary Examination for Metals (387) :—

Simple.			*Complex.*	
NH_4Cl $HgCl_2$ As_2O_3 KNO_3 Cr_2O_3	$Pb(NO_3)_2$ $NaCl$ $BaCl_2.2H_2O$ $Sr(NO_3)_2.4H_2O$ $CuSO_4.6H_2O$ (See note.)	$ZnSO_4.7H_2O$ $MnCl_2$ $Co(NO_3)_2.6H_2O$ $NiSO_4.7H_2O$ SnO_2	$AmCl+NaCl$ $HgCl_2+BaCl_2.2H_2O$ $MnCl_2+KCl$ $NiSO_4.7H_2O+$ $ZnSO_4.7H_2O$	$AlK(SO_4)_2.12HO$ $SnO_2+Sr(NO_3)_2.4H_2O$ $CaCO_3+ZnSO_4.7H_2O$ $Cr_2O_3+MnCl_2+$ $NaCl$

Note.—Best finely powdered, since its colour is then almost destroyed.

For the Preliminary Examination for Acid-radicles (410) :—

Simple.			*Complex.*
$CaCO_3$ $Na_2SO_3.7H_2O$ $CaCl_2O$ $NaCl$	FeS KNO_3 $KClO_3$ KI	$Na_2S_2O_3.5H_2O$ KBr $Na\overline{A}$	$CaCO_3+KNO_3$ $CaCl_2O+CaF_2$ Na_2CO_3+KI

A few of the above mentioned solids may then be examined by both Preliminary Examinations for both Metals and Acid-radicles.

539. Substances to be analysed by the General Course (380 *et seq.*).

The following lists are so arranged that the analysis of the substances contained in them is more difficult in each column proceeding from left to right, and usually also in proceeding in one and the same column from top to bottom. For examples of Alkaline solutions, see (537) :—

SUBSTANCES FOR ANALYSIS BY THE GENERAL COURSE.

1. Simple soluble substances.	2. Complex soluble substances.	3. Complex soluble substances.
$BaCl_2.2H_2O$ $MnCl_2$ $HgCl_2$ $MgSO_4.7H_2O$. KCl $NiSO_4.7H_2O$ *$CaCO_3$ $Pb(NO_3)_2$ *As_2O_3 Fe_2Cl_6 NH_4Cl $NaCl$ &c., &c.	$CaCl_2.6H_2O+BaCl_2.2H_2O+Sr(NO_3)_2.4H_2O$ $AlK(SO_4)_2.12H_2O+CrK(SO_4)_2.12H_2O$ $MnCl_2+ZnSO_4.7H_2O+CuSO_4.6H_2O$ $Pb(NO_3)_2+HgCl_2+NaCl$ *$As_2O_3+CaCO_3+BaCO_3$ *$ZnO+MgCO_3+KClO_3$ *$MgCO_3+BaCO_3+PbCO_3$ &c., &c.	Fe_2Cl_6 $MnCl_2$ $ZnSO_4.7H_2O$ KCl As_2O_3 $CrK(SO_4)_2\ 12H_2O$ $Co(NO_3)_2.6H_2O$ $Hg_2(NO_3)_2.H_2O$ $Sr(NO_3)_2.4H_2O$ KNO_3 $Bi(NO_3)_3.5H_2O$. &c., &c.

4. Substances yielding a Phosphate precipitate in Group III.	5. Substances partly or entirely insoluble.	
$CaCl_2.6H_2O$ Fe_2Cl_6 $Na_2HPO_4.12H_2O$ $BaCl_2.2H_2O$ $MgCl_2$ Fe_2Cl_6 $Na_2HPO_4.12H_2O$ $Co(NO_3)_2.6H_2O$ $CaCl_2.6H_2O$ $MnCl_2$ $CrK(SO_4)_2.12H_2O$ $Na_2HPO_4.12H_2O$ &c., &c.	$BaSO_4$ SiO_2+S $BaSO_4+CaF_2+SnO_2$ $BaCl_2.2H_2O$ $AlK(SO_4)_2.12H_2O$ $AgNO_3$ SiO_2 S C $PbSO_4+BaSO_4$ $PbCrO_4$, ignited.	*Metallic substances* (467 *et seq.*) Iron pyrites (FeS_2) Iron filings (Fe) Zinc clippings (Zn) Brass filings (Cu+Zn) German silver (Cu+Zn+Ni) Bronze (Cu+Sn) Type-metal (Sb+Sn+Pb) *Silicates* (480)— Fire-clay ($Al_2O_3.2SiO_2$) Brown-clay (do. +Fe) Window-glass (Na, Ca, SiO_2) Flint-glass (Pb, K, SiO_2) *Cyanogen Compounds* (482)— Any of the foregoing which has been mixed with KCy, KCyS, $K_4FeCy_6.3H_2O$, $K_6Fe_2Cy_{12}$, $K_6Co_2Cy_{12}$

* To be given in the solid state.

APPENDIX I.

REACTIONS FOR THE RARER ELEMENTS.

THIS Appendix contains some of the most important reactions for many of the rarer elements. The arrangement here used is that adopted in Section IV., those elements being placed together which are precipitated in the same analytical group: the groups, however, are arranged in the order in which they occur in the General Table. A scheme for the detection of these elements follows (553), showing in which group they are precipitated in the General Table, and by which reactions they will be most readily found.

GROUP I.—SILVER GROUP.

In this group are included Tl and Wo: the former is partially precipitated as chloride by HCl, the latter completely as tungstic acid.

Thallium is only partially precipitated in Group I. since its chloride is not quite insoluble in water: it belongs also to Group III.B, being entirely precipitated by Am_2S.

540. THALLIUM (Tl).—Use Tl_2SO_4 solution.

Tl occurs in small quantity in many natural sulphides, often also in the ashes of plants and in mineral waters, Tl yields both thall*ic* and thall*ious* salts, but the former are very unstable, changing even when their solutions are heated into thall*ious* salts.

HCl: a white precipitate, which rapidly settles, does not blacken in the light, and is soluble in aqua regia. It is soluble in a large quantity of water, and therefore does not form in dilute solutions.

KI: a yellow precipitate; almost insoluble in water, more soluble in KI solution. In a solution containing Fe, any ferric salt must first be reduced by H_2SO_3 before adding KI.

$PtCl_4$: orange-red precipitate, slightly soluble in water.

Am_2S: black precipitate, which is easily coagulated by heat, is insoluble in AmHO, alkaline sulphides, and in KCy: it is readily oxidised by the air to Tl_2SO_4, and is easily soluble in mineral acids. Tl is entirely precipitated by H_2S from a solution in which $H\bar{A}$ is the only free acid present, but free mineral acids prevent the precipitation entirely.

Flame coloration.—Thallium compounds impart to the Bunsen flame an intense *green* colour, which, however, rapidly disappears. The spectrum (p. 341) is very characteristic, consisting of one bright emerald-green line. Thallium may be easily detected by the spectroscope in solution, or better in any of its precipitates mentioned above.

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541. TUNGSTATES.—Use Na_2WoO_4 solution.

Wo usually occurs in the form of a tungstate. The insoluble tungstates yield soluble alkaline tungstates on being fused with alkaline carbonates. From a solution of alkaline tungstate HCl precipitates the tungstic acid entirely.

HCl, HNO_3, or *H_2SO_4:* white precipitate (H_2WoO_4), becoming yellow on boiling: insoluble in excess of acid, but soluble in AmHO. A piece of Zn dropped into the acid liquid containing the precipitate yields a deep blue colour.

$SnCl_2$ in neutral solution, made by dissolving $SnCl_2$ crystals in water and filtering: *yellow* precipitate, becoming *blue* on addition of HCl and heating.

Am_2S yields no precipitate in a solution of alkaline tungstate; but if, after adding Am_2S, the liquid is made acid with HCl, brown WS_3 is precipitated.

Microcosmic bead:—

In outer flame—*Colourless* or *yellow.*

In inner flame—*Blue:* if a little $FeSO_4$ is fused into the bead the colour changes to *blood-red.* These colours are best seen when the bead is perfectly cold.

GROUP II.A.—COPPER GROUP.

In this group are included Pd (Os, Rh, Ru): they are precipitated as sulphides by H_2S from acid solutions, and the sulphides are insoluble in Am_2S, and in caustic alkali solution.

542. PALLADIUM (Pd).—Use $PdCl_2$ solution.

Pd occurs as a metal in native platinum, also in gold and silver. Palladium solutions are reddish-brown, or yellow if dilute : addition of water precipitates a basic salt, unless sufficient free acid is present to prevent it.

H_2S: black precipitate, in neutral, alkaline, and acid solutions: insoluble in Am_2S, but soluble in boiling HCl or in aqua regia.

AmHO: flesh-coloured precipitate ($PdCl_2.2NH_3$); soluble in excess of AmHO to a colourless liquid, from which HCl precipitates yellow crystalline paladammonium-chloride ($N_2H_6Pd''Cl_2$).

$HgCy_2$: yellowish-white gelatinous precipitate ($PdCy_2$), slightly soluble in HCl, easily soluble in AmHO. Very characteristic reaction.

KI: black precipitate (PdI_2). Very characteristic.

GROUP II.B.—ARSENIC GROUP.

In this group are included Mo, Se, Te (Ir),

543. MOLYBDATES (Mo). Use Am_2MoO_4 solution.

Mo occurs as molybdate ; also as sulphide, which may be readily converted into MoO_3 by ignition in the air or by heating with HNO_3. Unignited MoO_3 dissolves in acids; ignited MoO_3 is insoluble in acids, but easily soluble in alkalis.

HCl, HNO_3, or *H_2SO_4,* added in small quantity to an aqueous solution of a molybdate, yields a precipitate which is readily soluble in excess of the acid.

H_2S, added in *very small* quantity to the acidified solution, gives a *blue* liquid, in larger quantities a *brown* precipitate (MoS_3) ; the precipitation becomes complete only when the solution is heated and H_2S passed for some time : the precipitate is soluble in solutions of alkaline sulphides and hydrates, from which it is reprecipitated by acid added in excess.

Zn or *$SnCl_2$,* added to a solution of a molybdate in HCl, colours it *brown, green,* or *blue,* according to its state of concentration.

KCyS, added to a solution acidified with HCl, gives no coloration ; but on dropping in a piece of Zn a beautiful *crimson* colour is produced, which, when the liquid is shaken with ether, is taken up by the ether.

Na_2HPO_4, added in very small quantity to a solution of a molybdate, acidified with HNO_3, gives, on gently warming the liquid, a yellow precipitate readily soluble in excess of alkali-hydrate solution.

Borax-bead: outer flame, *yellow ;* inner, *dark-brown.*

Microcosmic-bead: outer and inner flames, *green.*

544. SELENIUM (Se).—Use a metallic selenide, an alkaline selenite and selenate.

Se occurs as metallic selenides, *e.g.* of Fe, Cu, Ag. A selenide heated in an open tube evolves a smell of decaying horse-radish, and produces a grey or red sublimate of Se: the smell is very characteristic.

a. Selenites:—

H_2S gives in acid solutions, if cold a *yellow*, if hot a *reddish-yellow*, precipitate: soluble in Am_2S.

$BaCl_2$ in neutral solutions a white precipitate: soluble in HCl or HNO_3.

$SnCl_2$ or H_2SO_3 gives in the presence of free HCl a red, or in warm solutions a grey precipitate of Se.

Cu in a hot HCl solution becomes covered with a black film: the liquid on standing with the Cu for some time becomes coloured red with Se.

b. Selenates:—

HCl produces no change in the cold: but on boiling, Cl is given off and the selenate is reduced to selenite, to which the above tests under (*a*) may be applied.

$BaCl_2$: a white precipitate ($BaSeO_4$); insoluble in cold HCl, dissolved by boiling with HCl with evolution of Cl and reduction to $BaSeO_3$.

c. Selenium in any form of combination is detected by the following reactions:—

Heated on charcoal in the inner blowpipe flame a smell of rotten horse-radish is perceived.

Fused on charcoal with Na_2CO_3 in the inner blowpipe flame, a fused mass is obtained, which when moistened on Ag yields a black stain, and on addition of HCl evolves H_2Se, a badly smelling gas.

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545. TELLURIUM (Te).—Use a metallic telluride, an alkaline tellurite and tellurate.

Te occurs united with metals, *e.g.* Au, Ag, Bi, Cu, Pb.

A telluride heated in an open glass tube gives white fumes and a sublimate which differs from that given by Sb, by being fusible before the blowpipe.

a. Tellurites:—

H_2O: on dilution with water tellurous acid is precipitated from the acid solution.

H_2S: in acid solutions a brown precipitate (TeS_2); easily soluble in Am_2S.

H_2SO_3, $SnCl_2$, or *Zn* precipitates black Te.

b. Tellurates.—HCl produces no change in the cold; but on boiling, Cl is evolved and the tellurate is reduced tellurite; the solution then gives the above reactions under (*a*).

c. Tellurium in any form of combination, if fused with Na_2CO_3 on charcoal in the inner blowpipe flame, gives sodium telluride; a solution of which gives a black stain on Ag, and on being acidified deposits black Te and evolves H_2Te.

GROUP III.—IRON AND ZINC GROUPS.

In this Group are included U, In, Ti, Be, Tl [V], (Zr, Ce, Ta, Nb, La, Di, Y, E, Th).

Tl is often partially precipitated as chloride in Group I., and its reactions are given under that group. V is not precipitated by Am_2S unless acid is added in excess after Am_2S.

546. URANIUM (U).—Use $(UO_2)''(NO_3)_2$.

U occurs in nature principally as *pitchblende* (oxide), also as *uranite* (a hydrated uranium-calcium phosphate) and *chalcolite* (hydrated uranium-copper phosphate).

AmHO, KHO, or *NaHO:* yellow precipitate insoluble in excess.

Am_2S gives in neutral solutions a *dingy-yellow* or *brown* precipitate of uranium oxysulphide, which is soluble in Am_2CO_3 (diff. from ZnS, MnS, FeS, &c.). The precipitate settles slowly unless AmCl is added; it is soluble in acids, even in $H\bar{A}$: on being heated with Am_2S in excess it is changed into uranous oxide and sulphur.

H_2S produces no precipitate in acid solutions.

Am_2CO_3, $KHCO_3$, or *$NaHCO_3$:* yellow precipitate easily soluble in excess; from this solution the uranium is reprecipitated by addition of NaHO or KHO, or by boiling (diff. from Fe).

K_4FeCy_6 gives in acid solutions a reddish-brown precipitate, which is distinguished from the similar one produced in a copper solution by dissolving in excess of AmHO to a *yellow* fluid.

$BaCO_3$: complete precipitation even in the cold.

Zn changes the *yellow* colour of acid solutions to *green*.

Borax and Microcosmic beads:—

Outer flame, *yellow;* inner flame, *green*.

547. INDIUM (In).

Alkali-hydrates precipitate a hydrate resembling Al_2Ho_6, insoluble in excess. Indium solutions are also precipitated by alkaline carbonates, by Na_2HPO_4, by boiling their neutral solutions with $Na\bar{A}$, by $BaCO_3$, and by alkaline oxalates.

H_2S gives no precipitate in strong acid solutions, in dilute and feebly acid solutions a little sulphide separates, in a solution containing no free acid but $H\bar{A}$ the metal is entirely precipitated as *yellow* sulphide: the precipitate is insoluble in cold, soluble in boiling, Am_2S; from the boiling solution *white* sulphide separates on cooling.

Am_2S, added after $H_2\bar{T}$ and excess of AmHO, gives a *white* precipitate, becoming yellow on treatment with $H\bar{A}$.

Flame coloration; bluish violet. The spectrum shows two very characteristic blue lines, which are brilliant, but very rapidly disappear when the chloride is employed: see p. 341.

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548. BERYLLIUM (Be).—Occurs as silicate in phenakite, and as silicate with Al-silicate in beryl and emerald.

Am_2S, AmHO, KHO, or *NaHO:* flocculent hydrate is precipitated resembling Al_2Ho_6 in its appearance, and in being soluble in KHO. Differs from Al_2Ho_6 in being precipitated from its solution in KHO by dilution and long boiling, and by being soluble when freshly precipitated if it is long boiled with AmCl solution.

Alkaline carbonates precipitate a carbonate soluble in excess, especially in Am_2CO_3; from these solutions the carbonate is reprecipitated, on diluting and boiling for some time, with especial ease from the Am_2CO_3 solution (diff. from Al).

$BaCO_3$ precipitates Be-solutions completely: *$H_2C_2O_4$* and alkaline oxalates produce no precipitate. Moistened with $Co(NO_3)_2$ solution and heated on charcoal in the outer blowpipe flame, a *grey* mass is obtained (diff. from Al.

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549. TITANIUM (Ti).—Occurs as TiO_2, with traces of Fe, Mn, Cr, in rutile, anastase, and brookite: also as TiO_2 in combination with Fe in titaniferous iron ore.

Ignited TiO_2 is insoluble in water and most acids: it is easily soluble in HF, less readily in boiling strong H_2SO_4: it is also rendered soluble in cold water by fusion with $KHSO_4$.

TiO_2 differs from SiO_2 in not being volatilised when heated in a platinum dish with HF and strong H_2SO_4.

By dilution and long boiling, white flocculent hydrated TiO_2 is precipitated from solutions in H_2SO_4 or HCl, and from the aqueous solution of the fusion with $KHSO_4$: the precipitate is metatitanic acid : it cannot be filtered off unless an acid or AmCl is added.

AmHO, KHO, NaHO, Am_2S, or $BaCO_3$, white flocculent precipitate insoluble in excess : if precipitated and washed in the cold it dissolves in HCl and in dilute H_2SO_4.

Zn or *Sn* gives in acid solutions a *blue*, or if dilute a *rose-coloured*, liquid.

$Na_2S_2O_3$, on boiling, precipitates Ti-solution entirely (diff. from Fe).

Microcosmic bead:—

In the outer flame : *yellow*, whilst hot ; *colourless*, cold

In the inner flame: *yellow*, hot ; *violet*, cold. The production of these colours is much aided by adding a fragment of Sn. Addition of a small quantity of $FeSO_4$ gives in the inner flame a *blood-red* bead.

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550. VANADIUM (V).

Occurs in vanadinite [$3Pb_3(PO_4)_2.PbCl_2$], and in certain Fe- and Cu-ores.

Vanadium is known in several stages of oxidation ; it will usually occur in analysis as vanadic acid or a vanadate, which in acid solution is of a yellow or reddish colour.

H_2S, H_2SO_3, or $H_2C_2O_4$ reduces acid solutions of a vanadate, and causes the colour of the solution to change to *blue:* with H_2S a deposit of sulphur also forms.

Am_2S, if added in excess, gives a brown liquid, in which an excess of acid causes a *brown* precipitate of V_2S_5 ; excess of Am_2S dissolves this to a reddish-brown liquid.

Zn gives in a very dilute solution acidified with H_2SO_4 and gently warmed, a *blue* colour, which changes into *green* and then *lavender-blue*.

Solid AmCl, added until the solution is saturated, precipitates Am_3VO_4, which is insoluble in saturated AmCl solution. Very characteristic reaction.

H_2O_2 when shaken up with an acid solution imparts a red colour, which is not removed by shaking with ether. This is a very delicate test.

Borax bead:—

In outer flame; *colourless*, or *yellow* if much V is present.

In inner flame; *green* hot and cold; if much V is present *brown* hot, and *green* cold.

GROUP V.—POTASSIUM GROUP.

In this group are included Li, Cs, Rb.

551. Lithium (Li).

Li occurs frequently in mineral waters and in the ashes of plants, also in small quantities in the minerals lepidolite, triphane, and petallite. It is allied to the Barium Group by the difficult solubility of its carbonate and phosphate; and it differs from K and NH_4 by not being precipitated by $PtCl_4$ or by $H_2\overline{T}$: from Na it is readily distinguished by the flame coloration and spectroscope. LiCl is separated from KCl and NaCl by its insolubility in a mixture of absolute alcohol and ether.

Na_2HPO_4 added to the not too dilute solution, made decidedly alkaline with NaHO, gives, on boiling, a white crystalline precipitate (Li_3PO_4) which quickly settles. Traces of Li may be precipitated by adding Na_2HPO_4, then NaHO until the liquid remains alkaline, evaporating to dryness and washing the residue with dilute AmHO. This precipitate differs from the phosphates of Ba, Sr, Ca, and Mg by fusing in the blowpipe flame and being absorbed by the charcoal support, also by its diluted solution in HCl giving no precipitate on addition of AmHO in excess in the cold, but a white crystalline precipitate on boiling.

Flame coloration: this is *carmine-red*; the spectrum (see p. 341) consists of two lines, an intense carmine-red line (α) and a feeble orange-yellow line (β). This coloration is concealed by that of Na, which, however, does not interfere with the spectrum: the Na coloration is also removed if the flame is viewed through the indigo-prism, whereas that of Li can pass through the thinner parts of the prism; it differs from the K coloration, however, in being unable to penetrate to thick layers of indigo-solution or in being much weakened by its passage. As silicate, Li only gives the flame coloration after fusion with $CaSO_4$; the phosphate requires to be first moistened with HCl.

552. Rubidium (Rb), and Cæsium (Cs).

These metals occur in small quantities in some mineral waters and in a few minerals. Their compounds resemble those of

K in being precipitated by $PtCl_4$ and by $H_2\overline{T}$, and in giving a similar flame coloration. Characteristic differences are, the much greater insolubility in water of the platino-chlorides, which enables the K_2PtCl_6 to be dissolved away from Rb_2PtCl_6 and Cs_2PtCl_6 by boiling the precipitate with successive small quantities of water: the alums also show a similar difference in solubility in cold water.

Cs and Rb are readily distinguished from one another and from other elements by their spectra (see p. 341). For Cs the blue lines (α, β) are especially distinct and characteristic: in the Rb spectrum the indigo-blue lines (α, β) are very distinct, but the red lines (γ, δ) are most characteristic. For the spectroscopic test the chlorides are most suitable.

553. General Group Tabl

Group I.—Reagent HCl.	*Group II.*—Reagent H_2S.
The group pp. may contain in addition to $AgCl$, Hg_2Cl_2 $PbCl_2$:— *$TlCl$ (white).* *H_2WO_4 („).* *TlCl* will usually be readily detected by a spectroscopic examination of the group pp. It may be removed from the group pp. by boiling with a little water, and pp$^{d.}$ from the cold filtrate by KI (Pb is also thus pp$^{d.}$): the pp. is yellow, and gives the thallium spectrum. *H_2WO_4*: a white pp. becoming yellow on boiling. Its presence is confirmed by dropping a piece of Zn into a portion of the acid liquid and pp., when a *deep blue* colour will be obtained. Also by fusion of the pp. in a microcosmic bead, which will be *colourless* or *yellow* in the outer flame, *blue* (or with $FeSO_4$, *blood-red*) in the inner flame.	*The group pp. may contain as sulphides* *addition to Hg, Pb, Bi, Cu, Cd :—* *Pd (black-brown)* } *Tl (black; pp$^{d.}$ with As_2S_3)* } Insolub *[Os, Rh, Ru.*]* } Am *And in addition to SnS, SnS_2, Sb_2S_3, A* *Au_2S_3, PtS_2 :—* [*Ir*-sulphide*] } *Mo-* „ (*brown*) } Soluble in *Se-* „ (*red-yellow*) } Am_2S. *Te-* „ (*black*) } The *yellow* colour of a solution containi is changed to *blue* by H_2S. *Tl* is readily found by the spectroscope group pp. *Pd* will remain in solution in excess of with Cd and Cu in Table II.A: from this it is pp$^{d.}$ by adding HCl: its presence further confirmed by the $HgCy_2$ or (542). The examination of the sulphides in Gro when the above elements may be presen commenced by fusion with Na_2CO_3 and From the fused mass, water dissolves arsenate, -molybdate, -selenate, and -te leaving SnO_2, sodium-antimonate, Au, Ir undissolved; the elements may then tected by special tests. For the separati detection of the platinum metals a larg must be consulted. * Only completely pp$^{d.}$ if the liquid ha warmed, and H_2S passed for a long time.

TATION OF RARER ELEMENTS.

III.—Reagents AmCl, AmHO, and Am_2S.	Precipitated from the filtrate from Group III. by excess of HCl.	*Group V.*
oup pp. may contain lphides, in addition to n, Mn, Ni, Co:— (*black brown.*) *n*(,, ,,) *l*(,, ,,) *s hydrates in addition l, Cr* (*and phosphates,*)— *ite, flocculent.*) *lourless, gelatinous*). *Nb, Ce, La, Di, Y, E,* d *In* will usually be ily detected by a spec- opic examination of the y be first separated and cted by dissolving some he group pp. in boiling te HCl, and reducing Fe present in the solu- by H_2SO_3; then pre- tating Tl by addition of and testing the pp. by spectroscope. re complete scheme for analysis of this group will be found in par. (pp. 336, 337).	*As sulphides*:— *V* (*black*). *W* (trace, *brown*). *Ni* (trace, *black*). This pp. is dried and then fused with a mixture of Na_2CO_3 and KNO_3: on boiling the mass with water, NiO remains undissolved; it is filtered off, and its presence confirmed by fusion in a borax bead. The aqueous solution may contain alkaline vanadate and tungstate. V is separated by saturating the liquid with AmCl, and its presence confirmed in the pp. by the borax bead, and by dissolving the pp. in HCl and employing the reactions with H_2O_2 and with Zn (550). The filtrate, after concentration if necessary, is acidified with HCl, and the presence of W confirmed by the Zn reaction and the microcosmic bead (541.)	The filtrate after the separation of all the group precipitates may contain besides Mg, K, and Na:— *Li, Cs, Rb.* These metals are readily detected by their very characteristic spectra (p. 341). LiCl may also be detected by its behaviour with Na_2HPO_4. Before proceeding to the spectroscopic examination, it is well to convert them into chlorides if they are not already in that form. If the chlorides are evaporated to dryness and extracted with absolute ether and alcohol, to which a few drops of HCl have been added, LiCl, RbCl, CsCl dissolve, whilst the greater part of the KCl and NaCl remains undissolved. The solution is evaporated to dryness, dissolved in a little HCl, and Rb and Cs are pp$^{d.}$ by $PtCl_4$: this pp. is boiled with small quantities of water until it no longer gives the K spectrum, the spectra of Rb and Cs will then be seen, if present. Li is found in the filtrate after adding $PtCl_4$.

554.—Table for Analysis of Group III. Precipitate,

The precipitate may contain Fe, U, In, Tl, Al, Cr, Zn,

<table>
<tr><td colspan="4">After a preliminary spectroscopic examination has been made of a small portion of the the General Table (553), the group pp. is dried and fused in platinum with $KHSO_4$ for long time with cold water:—</td></tr>
<tr><td rowspan="6"><i>Residue:</i> may contain Ta_2O_5, Nb_2O_5, also SiO_2, and traces of Fe and Cr which have escaped solution. By fusion with $KClO_3$ and NaHO, Cr and Si are rendered soluble in dilute NaHO; from the residue, insoluble in NaHO, Ta_2O_5 and Nb_2O_5 may be removed by washing with dilute Na_2CO_3 solution.</td><td colspan="3"><i>Solution:</i> Reduce Fe by adding H_2SO_3 in excess, dilute consider covering the vessel, and by passing a stream of CO_2 through it,</td></tr>
<tr><td rowspan="5"><i>Pp.</i> may consist of <i>TiO_2</i>, possibly also traces of Zr.
Confirm the presence of TiO_2 by the microcosmic bead (549).</td><td colspan="2"><i>Filtrate:</i> add a few drops of strong HNO_3, and precipitate once more by adding excess</td></tr>
<tr><td colspan="2"><i>Pp.</i> may contain Fe, In, U, Cr, Al, Be, &c.</td></tr>
<tr><td colspan="2"><i>Pp.</i> may contain Fe, In, U, &c. Dissolve large excess of HCl, add $BaCO_3$, and let</td></tr>
<tr><td rowspan="2"><i>Pp.</i> may contain Fe, In, U, also traces of Al and Cr.
It is dissolved in HCl, and $NaHCO_3$ is added in excess, whereby U alone is obtained in solution.
In is found by the spectroscope.
Cr by fusion with Na_2CO_3 and $KClO_3$.</td><td><i>Filtrate</i> may contain Th. Ba is pp^{d} by H_2SO_4, the liquid exactly neutralised of K_2SO_4 are added, allowed to stand for pp. is washed with</td></tr>
<tr><td><i>Pp.</i> may contain Zr, Th, Ce.
Th and Ce are dissolved away by boiling with dilute HCl, and are repp^{d} by AmHO.</td></tr>
</table>

WHEN THE RARER ELEMENTS MAY BE PRESENT (FRESENIUS).

Mn, Ni, Co, Ti, Be, [Zr, Ta, Nb, Ce, La, Di, Y, E, Th].

pp. for Tl and In, and Tl has been further tested for, if necessary, by KI as directed in
some time, the cold mass is then powdered and allowed to stand, with shaking, for a

ably and boil for a long time, preserving the liquid from oxidation by the air by
unless it smells constantly of SO_2 :—

concentrate by evaporation, and add AmHO in excess : filter, dissolve the pp. in HCl,
of AmHO :—

Dissolve it in HCl, and add excess of cold strong KHO solution :—		*Filtrate* may contain :— Zn, Mn, Ni, Co.
in HCl, boil off any stand :—	*Filtrate* may contain Al, Cr, Be : dilute and boil for some time, filter. Al remains in solution, and may be pp^d by adding HCl just in excess, then AmHO in excess. Cr and Be are separated by fusion with Na_2CO_3 and $KClO_3$, dissolving in water and pp^g.; Be by adding HNO_3 in excess, then AmHO in excess.	
Zr, Ce, La, Di, Y, E, adding excess of is concentrated and with KHO. Crystals and the liquid is some hours. The K_2SO_4 solution.		
Sol^n may contain Y, E, also Be. The solution is pp^d by AmHO, and Be dissolved away from it by $H_2C_2O_4$.		

APPENDIX II.

USE OF THE SPECTROSCOPE FOR ANALYSIS.

555. When the light from a bright gas or candle-flame is allowed to pass through a narrow slit, and is then looked at through an appropriately cut glass prism, a series of colours is seen following one another without break or interruption; such a band of colours has received the name of a "continuous spectrum." The direct-vision spectroscope* is a little instrument furnished at one end with the requisite slit, whose width may be varied by means of a projecting screw-collar: at the other end will be found a draw-tube containing lenses for focussing, and the body of the instrument incloses the glass prisms. On looking at a bright gas or candle-flame through this spectroscope a "continuous spectrum" will be seen, whose margins may be sharply focussed by means of the draw-tube. A similar spectrum is seen when any brightly glowing *non-volatile* substance is viewed through the spectroscope.

Many substances, however, when strongly heated, volatilise; and their *glowing vapours* emit light whose colour is in many cases peculiar and characteristic: mention has been already made of this fact in par. 32, and its importance as a means of detecting certain elements was there stated.

When such a vapour is viewed through the spectroscope, a "discontinuous or "line spectrum" is seen, which consists of one or more coloured "lines" or "bands," whose position and colour are characteristic of that particular vapour. Thus Na vapour yields one bright yellow line, Li one intense carmine line, whilst K gives two lines, one red and the other violet. These lines are readily seen by holding in the Bunsen flame a loop of platinum wire, which has been previously dipped into strong NaCl, LiCl, or KCl solution, and viewing the coloured flame through the spectroscope. But not only does this method of analysis serve to detect the elements when occurring separately, it also detects each of them when two or more are present together, since the spectrum of each is then seen without any interference from the others: thus although the *flame coloration* of K or of Li is entirely masked by that of Na when the chlorides of both metals are held in the flame together, the two *spectra* are distinctly seen side by side when the flame is looked at through the spectroscope; and both Li and K are thus readily recognised when present with Na.

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* This is the cheapest and most handy form of spectroscope, and may be purchased of the makers, Messrs Browning, of the Strand, London.

The delicacy of this method of detection is extreme; thus the $\frac{1}{3,000,000,000}$th of a gram of Na can be distinguished by the appearance of its yellow spectral line.

The accompanying chart (p. 341) gives the relative position of the most important lines in eleven different spectra. It is meant to serve merely as a guide to the beginner: when the student has accustomed himself to the appearance of the spectra, reference to the chart will become unnecessary.

It is important to remember that the coloration of the Bunsen flame can only be produced by substances which are volatile at the temperature of the flame: and that the coloration due to any element is more intense the more volatile is the compound employed. Thus KCl and KNO_3 are readily volatilised in the flame, and colour it strongly; K_2SO_4 is somewhat less volatile, and the coloration it yields is correspondingly feeble; K_2CO_3 is still less volatile, and imparts little colour to the flame; whilst K_2SiO_3 is non-volatile, and yields no flame coloration whatever. Hence in testing for an element by the spectroscope, care must be taken to ensure its conversion, if present, into a form of combination which is volatile in the Bunsen flame. As a rule, chlorides and nitrates of the metals are the most volatile salts, sulphates are less volatile, carbonates still less so, and silicates are usually non-volatile. It is in many cases sufficient to moisten the substance upon the platinum wire loop with strong H_2SO_4 or HCl before introducing it into the flame; the sulphates of Ba, Sr, and Ca should be first reduced to sulphides by heating them in the inner blowpipe flame, the sulphides are then readily converted into chlorides by moistening them with HCl. K_2SiO_3 in a natural silicate yields no K flame coloration; by fusion of the powdered mineral with gypsum powder, however, the K_2SiO_3 is converted into K_2SO_4, and the sulphate yields the K-coloration readily:—

$$K_2SiO_3 + CaSO_4 = K_2SO_4 + CaSiO_3.$$

Owing to the invariable presence of small quantities of Na in atmospheric dust, and to the extreme delicacy of the spectroscopic reaction for Na, the Bunsen flame always shows the Na line when looked at through the spectroscope, and the brightness of the line may be increased by striking the table, or in any other way raising dust, in the neighbourhood of the flame. This is in several respects advantageous; since, by rendering the Na line distinct by regulating the width of the slit and the position of the focussing lens, the spectroscope may be placed in adjustment before introducing into the flame the substance whose spectrum is to be observed. The Na line further serves as a fixed point from which the position of the lines of other elements may be judged; and in order to assist the student in so using it, the position of the Na line is indicated in all the other spectra on the accompanying chart by means of a dotted line.

556. When working with the spectroscope, the Bunsen flame should be so placed as to have a dark background, in order that light *from the flame only* may enter the slit: if not working in a darkened room, a piece of black velvet or cloth may be hung behind the flame; this is, however, not indispensable, it will usually be sufficient to avoid the entrance of direct or brightly-reflected light into the instrument. It is best to view first either the sky, or a bright flame—such as that obtained by closing the air-holes at the foot of the Bunsen burner, and to turn the spectroscope into such a position that the continuous spectrum becomes horizontal with its red end to the left hand; the instrument is next directed on a portion of the non-luminous Bunsen flame about two-thirds of the height from the flame's base, and held either by a clamp, or very steadily by the hand, in this position. The Na line is then focussed as directed above: and, whilst carefully watching the flame through the spectroscope, the substance is introduced into the outer part of the front of the flame at about one-third the flame's height from its base: the observation of the flame is continued for a short time after moistening the substance with acid as directed above. The careful observation of the flame during the introduction of the substance into the flame is necessary, since some elements (*e.g.*, Tl), when present in small quantity, give a spectrum which appears only as a momentary flash: other substances appear only as the substance attains the full temperature of the flame, and their spectra are therefore seen only after the substance has been heated in the flame for some time.

The regulation of the width of the slit must depend upon circumstances. A wide slit admits more light and increases the brightness of the lines; a narrow slit causes the lines to be more widely separated, and thus prevents neighbouring lines from blending, and generally enables their position to be more accurately noted. It is well to use a narrow slit, unless working with a faint flame coloration, or unless lines of feeble intensity have to be looked for.

The spectroscope is more especially useful in qualitative analysis:—

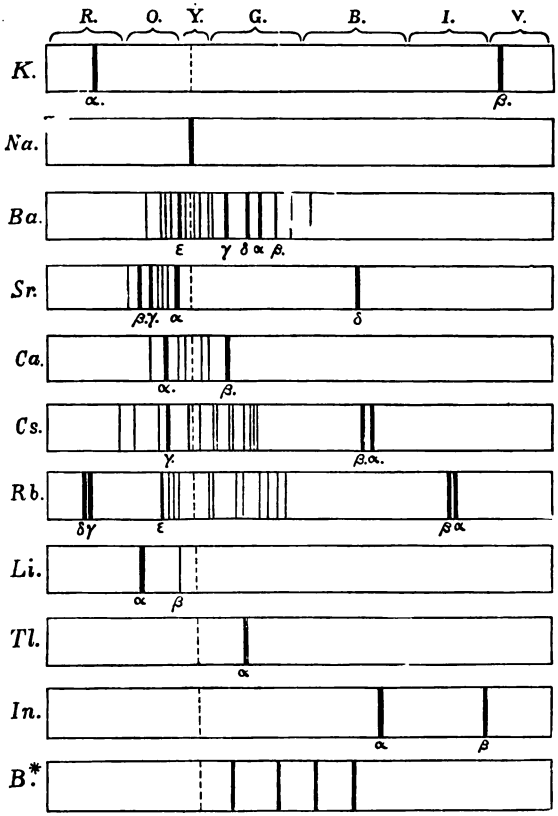
1st. In detecting readily K and Li, and other substances whose flame colorations are masked by that of Na, when occurring with Na.

2nd. It also rapidly distinguishes Sr from Ca; since as shewn in the chart the Sr spectrum contains a characteristic blue line δ, as well as certain red lines situated to the left hand of all those in the Ca spectrum; the green line β of Ca is also very distinctive of that element. Ba is specially characterised by the four green lines, α, β, γ, δ.

3rd. The spectroscope is of great value for detecting the rarer metals Li, In, Tl, Cs, Rb, which usually occur in comparatively small quantity in nature: the last four of these metals were first searched after and discovered on account of their very marked spectra having been seen by the spectroscope.

557. *Spectrum Chart.*—In using the chart below, the colour of a line in any spectrum may be found by referring to the head of the chart, and seeing in which of the bracketted spaces it falls; above each bracket is placed the initial letter of the name of the colour: the colours run from left to right in the following order:—Red, Orange Yellow, Green, Blue, Indigo, Violet.

The chart is a reduction from Bunsen's map: the relative position of the lines is given correctly: those in each spectrum which are most striking or characteristic are made thicker than the rest, and are denoted by Greek letters in the order of their relative importance. Many of the thin lines will not be seen by means of a small pocket spectroscope.



* This spectrum is seen when boric acid or a borate is fused with a little Na_2CO_3 on a loop of platinum wire, and the flame coloration obtained from this bead is examined.

APPENDIX III.

558. LIST OF CHEMICAL ELEMENTS, WITH THEIR SYMBOLS AND ATOMIC WEIGHTS.

Note.—The words in brackets are the Latin names of the elements from which the symbols have been derived.

Name.	Symbol.	Atomic Weight.	Name.	Symbol.	Atomic Weight.
Aluminium . .	Al	27·5	Molybdenum . .	Mo	96
Antimony (stibium)	Sb	122	Nickel . . .	Ni	59
Arsenicum . .	As	75	Niobium . .	Nb	94
Barium . .	Ba	137	Nitrogen . .	N	14
Bismuth . .	Bi	210	Osmium . .	Os	199
Boron . . .	B	11	Oxygen . .	O	16
Bromine . .	Br	80	Palladium . .	Pd	106
Cadmium . .	Cd	112	Phosphorus . .	P	31
Cæsium . .	Cs	133	Platinum . .	Pt	197
Calcium . .	Ca	40	Potassium (kalium)	K	39
Carbon . . .	C	12	Rhodium . .	Ro	104
Cerium . . .	Ce	92	Rubidium . .	Rb	85
Chlorine . .	Cl	35·5	Ruthenium . .	Ru	104
Chromium . .	Cr	52·5	Selenium . .	Se	79·5
Cobalt , . .	Co	59	Silicon . . .	Si	28
Copper (cuprum) .	Cu	63·5	Silver (argentum) .	Ag	108
Didymium . .	D	96	Sodium (natrium) .	Na	23
Erbium . . .	E	112	Strontium . .	Sr	87·5
Fluorine . .	F	19	Sulphur . .	S	32
Glucinum . .	G	9·5	Tantalum . .	Ta	182
Gold (aurum) .	Au	197	Tellurium . .	Te	129
Hydrogen . .	H	1	Thallium . .	Tl	204
Indium . . .	In	113·4	Thorinum . .	Th	238
Iodine . . .	I	127	Tin (stannum) .	Sn	118
Iridium . .	Ir	197	Titanium . .	Ti	50
Iron (ferrum) .	Fe	56	Tungsten (wolframium) .	W	184
Lanthanum . .	La	92			
Lead (plumbum) .	Pb	207	Uranium . .	U	120
Lithium . .	L	7	Vanadium . .	V	51
Magnesium . .	Mg	24	Yttrium . .	Y	62
Manganese . .	Mn	55	Zinc . . .	Zn	65
Mercury (hydrargyrum) .	Hg	200	Zirconium . .	Zr	89

559. THERMOMETRIC SCALES.

There are two different thermometric scales in use in this country, the Centigrade and Fahrenheit; the former of these is rapidly becoming universal for scientific purposes. The two scales are mutually convertible by the following formulæ, in which F.° represents a temperature on the Fahrenheit scale, C.° a temperature on the Centigrade scale:—

$$\left.\begin{array}{l} \frac{5}{9}(\text{F.}^\circ - 32) = \text{C.}^\circ \\ \frac{9}{5}\,\text{C.}^\circ + 32 = \text{F.}^\circ \end{array}\right\}$$

The temperatures occasionally referred to in this treatise are given on the Centigrade scale.

WEIGHTS AND MEASURES.

560. The corresponding values of the French and English weights and measures are here given; the use of the French or decimal system is strongly recommended by its extreme simplicity, since the smaller denominations are obtained by taking a tenth, hundredth, thousandth, &c., of the unit chosen, and are designated by the Latin prefixes deci-, centi-, milli-, &c., whilst the higher denominations are 10 times, 100 times, 1000, &c. times the unit, and are named by the Greek prefixes deca-, hecto-, kilo-, &c.; examples of this will be found in the tables given below.

561. The starting-point of the French system is the "metre" (=39·37 inches); this is the "unit of length." The "unit of measure" is the "litre," which is one cubic decimetre: the "unit of weight" is the gramme*, which is the weight of 1 cubic centimetre of distilled water at 4° C.

The chief conveniencies arising from the use of this system are:—

1st. That all the different denominations can be written as one, since they are either multiples by ten or are decimal fractions of the unit. Thus 5 decagrams, 3 grams, 4 decigrams, 8 milligrams, would be written 53·408 grams.

2nd. That since 1 cubic centimetre of water at 4° C. weighs 1 gram we may obtain the *weight* of water to be used from the *measure* by simply converting the measure into cubic centimetres, the number thus obtained will represent at once the corresponding weight of water in grams. Of course this conversion is strictly accurate only when the water is measured at 4° C., but for ordinary purposes the error introduced when the water is at the temperature of the air is too small to be of any importance in the preparation of solutions.

The weights and measures most frequently used for chemical purposes are the gram, the millimetre,† the litre, and the cubic centimetre‡ which is $\frac{1}{1000}$ of a litre.

MEASURES OF LENGTH.			ENGLISH.				
	metre.	inches.	mile.	furlong.	yards.	feet.	inches.
†Millimetre =	0·001 =	·03937 =					·03937
Centimetre =	0·01 =	·39371 =					·39371
Decimetre =	0·1 =	3·93708 =					3·9371
Metre =	1·0 =	39·37079 =				3	3·371
Decametre =	10·0 =	393·70790 =			10	2	9·7
Hectometre =	100·0 =	3937·07900 =			109	1	1
Kilometre =	1000·0 =	39370·79000 =		4	213	4	10·2
Myriometre =	10000·0 =	393707·90000 =	6	1	156	0	6

1 inch = ·0254 metre.
1 foot = ·3048 „

---

* Frequently written "gram" in English, for the sake of brevity.

† Usually written (m.m.).

‡ Usually abbreviated to (c.c.)

MEASURES OF CAPACITY.

1 litre = 4 cubic decimetre.

	litre.	cubic inches.	pints.
Millilitre, or Cubic centimetre (c.c.) =	·001	= ·06103	= 0·00176
Centilitre =	·01	= ·61027	= 0·01761
Decilitre =	·1	= 6·1027	= 0·17608
Litre =	1·0	= 61·027	= 1·76077
Decalitre =	10·0	= 610·27	= 17·60773
Hectolitre =	100·0	= 6102·7	= 176·07734
Kilolitre =	1000·0	= 61027·0	= 1760·77341
Myriolitre =	10000·0	= 610270·0	= 17607·73414

1 cubic inch = ·01639 litre.
1 cubic foot = 28·31531 litres.
1 gallon = 4·54336 „

MEASURES OF WEIGHT.

1 gram = the weight of 1 cubic centimetre (c.c.) of water at 4° C.

	grams.	grains.	Avoirdupois.		
Milligram	= ·001	= 0·01543			
Centigram	= ·01	= 0·15432			
Decigram	= ·1	= 1·54323			
Gram	= 1·0	= 15·43235	lbs.	oz.	drms.
Decagram	= 10·0	= 154·32349	= 0	0	5·65
Hectogram	= 100·0	= 1543·23488	= 0	3	8·5
Kilogram	= 1000·0	= 15432·34880	= 2	3	5
Myriogram	= 10000·0	= 154323·48800	= 22	1	2

1 grain = 0·0649 gram.
1 oz. (Troy) = 31·1035 grams.
1 lb. (Avoirdupois) = 453·593 „

ENGLISH WEIGHTS AND MEASURES.

Apothecaries Weight.

lb. oz. drms. scruples. grains.
1 = 12 = 96 = 288 = 5760
1 = 8 = 24 = 480
1 = 3 = 60
1 = 20

Avoirdupois Weight.

lb. oz. drms. grains.
1 = 16 = 256 = 7000
1 = 16 = 437·5
1 = 27·343

Imperial Measure.

gallon. pints. fluid oz. fluid drms.
1 = 8 = 160 = 1280
1 = 20 = 160
1 = 8

1 gallon = 70,000 grains of water at 16·7° C
1 fluid ounce = $\frac{1}{20}$ pint = 437·5 „

1 gallon = 277·280 cubic inches.
1 fluid ounce = 1·733 „

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